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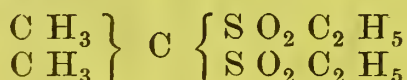
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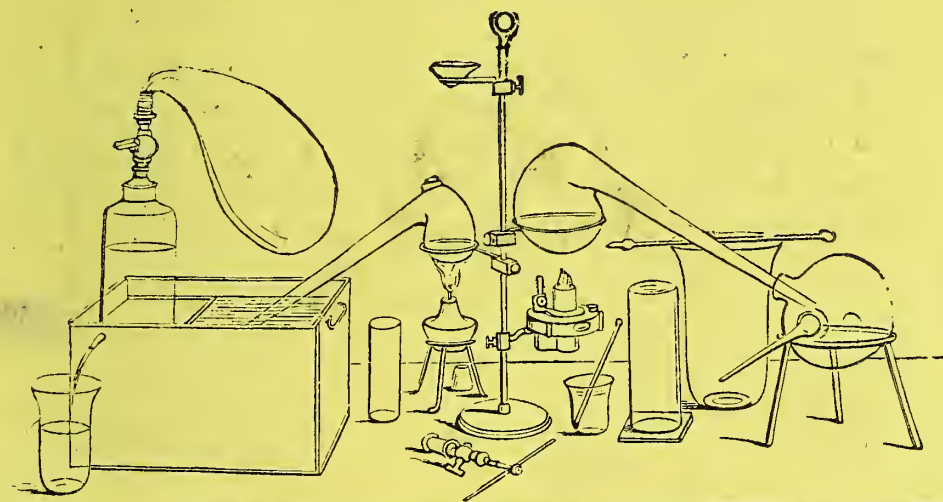
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Special Drugs and Chemicals  
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Ingredients of Pills and How  
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Lotions, Liniments, and In-  
jections  
Dispensing Foreign Prescrip-  
tions  
New Remedies  
Homœopathic Dispensing  
Illegible Prescriptions  
Examination Prescriptions  
Appendix [including French,  
German, and Latin Pre-  
scription terms, and poso-  
logical table]  
Index

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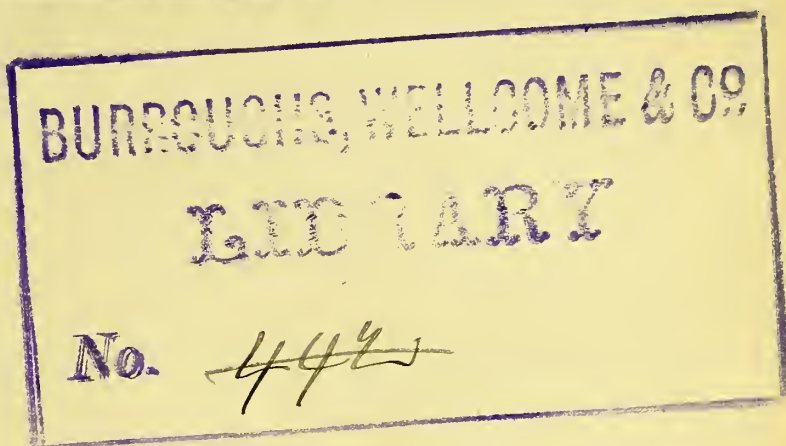
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PHARMACEUTICAL TESTING

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SPOTTISWOODE AND CO., NEW-STREET SQUARE  
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A MANUAL  
OF  
PHARMACEUTICAL TESTING

FOR THE  
MAN OF BUSINESS AND HIS ASSISTANTS

COMPRISING SIMPLE INSTRUCTIONS FOR THE TESTING OF THE  
CHEMICALS OF THE BRITISH PHARMACOPŒIA ETC. WITH  
SUCH MATERIALS AND APPLIANCES AS ARE IN  
COMMON USE AT THE DISPENSING COUNTER

BY  
BARNARD S. PROCTOR, F.I.C.

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No. PREFACE



EVERY pharmacist should test his chemicals occasionally, to see that their quality is up to the required standard. To do this frequently, the tests must be the simplest, speediest, and most inexpensive that may be devised. The tests should answer the question, Is the article fit for use? And, having given an answer to this, it is in many cases of little moment whether or not the extent of deviation from standard be indicated. It is usually better and simpler to reject that which is faulty than to make an exact analysis and allow for its deficiencies. Absolute purity is a thing only theoretically attainable, and it is better to define within clear and reasonable limits what may be allowed in practice than to give definitions which, from being unnecessarily and impracticably stringent, become a dead letter.

There is a wide distinction, which should be carefully noted, between the presence of those impurities, such as water, which only detract from the activity of a drug, and those which are active agents for evil, such as the presence of arsenium in the mineral acids. One per cent. of water above the legitimate proportion would scarcely trouble us, while one-tenth of this quantity of arsenium present as an impurity would totally unfit the sample for use.

In the following pages the attempt has been made to give

such directions for testing as will enable any ordinary pharmacist to examine and determine the quality of his pharmacopœial chemicals with only such reagents and appliances as are usually to be found at the dispensing counter, and by such operations as are in daily use in dispensing. The writer has endeavoured to point out the impurities likely to be present from faulty manufacture, and to these the tests most pointedly apply. Impurities, occurring as adulterations, must necessarily be more difficult to contend with, as an adulterator would naturally add only such impurities as would not be readily detected by the tests in ordinary use. Systematic volumetric work has not been included, as, to the ordinary pharmacist who does not keep volumetric apparatus and normal solutions always ready, it is more trouble to prepare for an operation of this kind than it is to obtain the required results by a gravimetric method. There are sundry cases in which any deviation from the B.P. tests would not be legitimate ; for example, the testing of opium and cinchona ; so long as it is required that a particular result is to be obtained by a particular process, there is no alternative but to follow that process. In other instances the B.P. tests are sufficiently simple and satisfactory, and there is no reason to deviate from them, but in a considerable number of cases there are described simpler methods of attaining the required indications, either as the direct result of experiments tried for the purpose, or the adoption of tests which others have described, and which have proved satisfactory in the writer's hands. For the benefit of young readers, comments of an explanatory nature have been made on the B.P. tests where that appeared necessary. Sundry of the official methods are more in the style of chemical exercises than guides to a business man as to how he should proceed to detect a suspected impurity, and, though the Pharmacopœia does not teach the lesson which



may be derived from its text, the student must not omit to learn it.

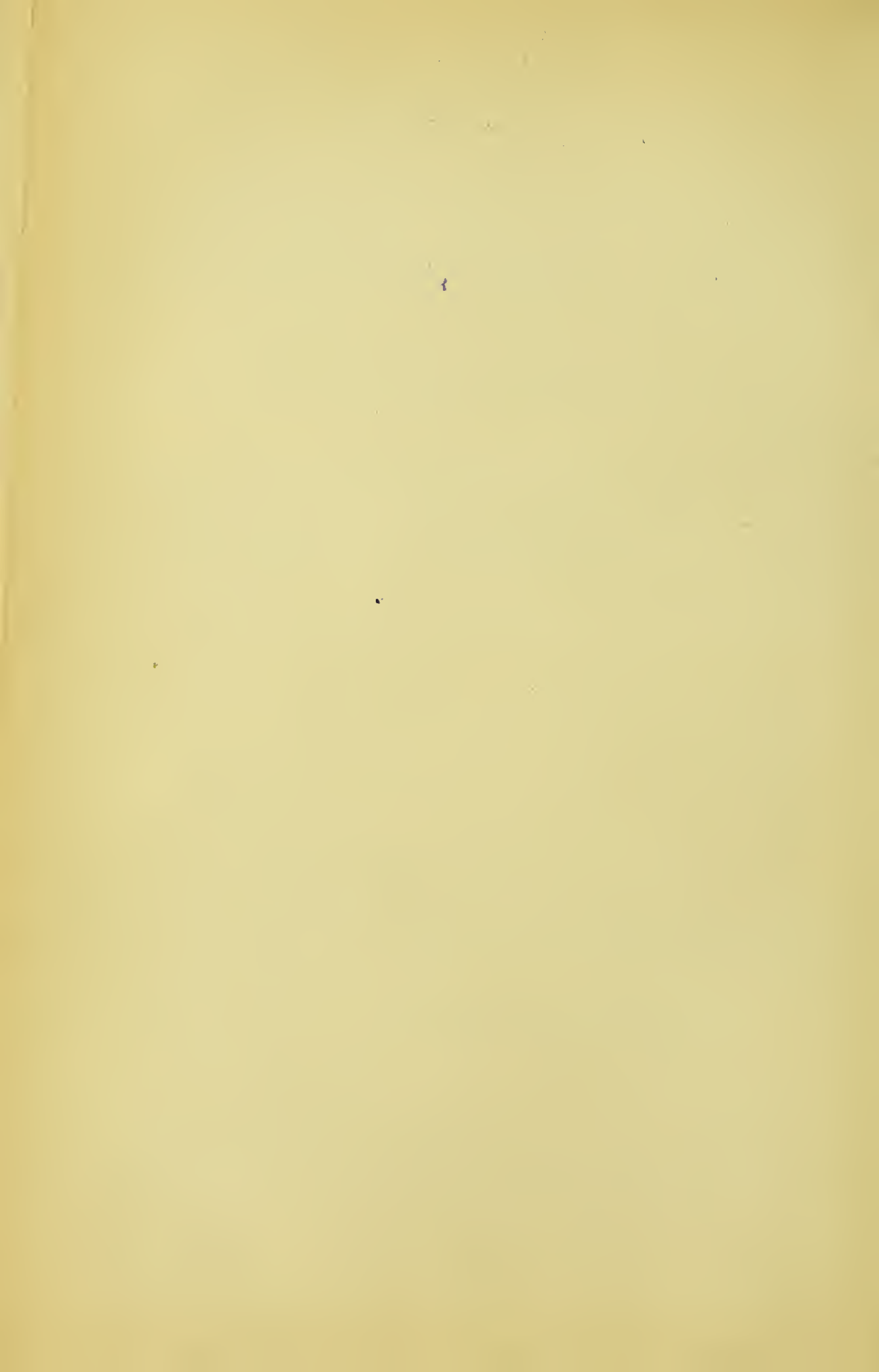
It has not been felt desirable in the present volume to treat of organised materials and their microscopic structures, but some articles of organic materia medica are noticed, which may be tested by methods analogous to those applied to trade chemicals.

A chapter on apparatus and manipulation has been introduced, to save the necessity of describing processes at length each time they occur.

It is hoped that this brief and imperfect handbook will encourage the habit of testing the chemicals used in medicine, and that the habit will in many cases be developed into a higher class of investigation.

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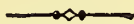
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No.

## A MANUAL

OF

# PHARMACEUTICAL TESTING



### *MANIPULATION, &c.*

**Weighing and Measuring.**—As the intention of the present little volume is to describe such analytical examinations as the dispenser may reasonably conduct at his dispensing counter, and by the aid of such appliances as may be expected to have their place there, a chapter on apparatus and operations will naturally be brief ; it will include only a few common-place matters, of which no analyst would need to be reminded, but which may be useful to such pharmacists as have not had the advantage of an analytical training.

It is assumed that the degree of accuracy which ought to be demanded in pharmaceutical testing is the same—neither more nor less than that accuracy which is demanded of the pharmacist in the performance of his dispensing and operative processes, and, in this view, good qualities of the usual pharmaceutical measures, weights, and balances, if carefully used, give results of a satisfactory degree of accuracy for the practical valuation of remedial activity.

It is not suggested that the dispenser's scales and weights are fine enough to estimate the quantity of lead and arsenium that may be present in an impure sample of sulphuric acid, but

that is quite unnecessary ; if the lead and arsenium are proved to be present, that is sufficient ground for the sample being rejected, without the trouble of their quantitative determination. The therapeutic valuation or assay of *the acid* is closely enough performed by ordinary measuring and weighing.

In weighing a sample of any drug for assay, it is desirable to weigh it in the condition in which it is kept for use, and, if it be required to determine the percentage of the active principle in the dry drug, to take a second portion in which to estimate the moisture.

Unstamped apothecaries' weights should not be trusted for any purpose without verification ; those bearing the Government stamp may be accepted.

Apothecaries' box beam scales usually *turn* with  $\frac{1}{10}$  grain, and with care will weigh a quarter of a grain fairly well ; those on pillar with lifting lever are preferable to hand scales, as they facilitate using one pan constantly for the weights ; and the German forms with long pendulous pointer and graduated scale have a further advantage, as they also facilitate the balancing to the same point on the scale, thus eliminating some sources of error.

Similar remarks may be made upon glass measures : those which are not stamped and not verified by personal examination, though frequently well-graduated, are not to be relied upon. The measurement should be taken from the general surface of the liquid (not from the capillary edge), and, the narrower the measure be in proportion to the bulk of the liquid under measurement, the less error is liable to be involved.

**Specific Gravity.**—A specific gravity bottle is upon the whole the most convenient and generally applicable means of testing the density of liquids.

In the British Pharmacopœia the legitimate density of liquids is frequently indicated in cases where the examination of the same is not worth the trouble, such as vinegar, hydrocyanic acid, or saccharated lime-water, but there are so many cases where the density is the readiest mode of getting im-

portant information that a gravity bottle ought to be in every pharmacist's hands. A hundred-grain bottle with its contents is about burden enough for the grain-scales, and will give results close enough for all the most important cases, as it will give the second decimal point accurately, and a close approximation to the third, while the British Pharmacopœia in some instances does not go beyond the second point, and never goes beyond the third.

In using the sp. gr. bottle it is convenient to have a counterpoise weight exactly balancing the dry bottle and its stopper, and another balancing the weight of water it will contain, or the weight of water *plus* the weight of the bottle: there will thus be less chance of errors, as fewer weights will be required in the operation.

It is also very convenient to have a selection of sp. gr. beads for fluids which are in constant use and require watching, such as the bead 838 for rectified spirit and 920 for proof.

**Solubility.**—The solubility of a material in water is constantly used as an indication of its quality. The statement may be that it should be totally soluble without any definition of the quantity of solvent, in which case the important point is to use enough of the solvent, and to aid its action, if slow, by trituration, to comminute the material under examination; the appearance of an insoluble residue in such cases being the indication of foreign matter. If the bulk dissolves freely and a little residue remains, which after decantation of the solution fails to dissolve freely in a little more of the solvent, it may generally be regarded as conclusive of the substance under examination being a mixture of two or more bodies, one of which is much less soluble than the bulk, and is presumably an impurity. On the other hand, if a body not very freely soluble is under examination, and is treated with a small portion of solvent, a definite measure of the saturated solution being evaporated to dryness, and the percentage of residue noted—if the undissolved portion, on being again treated in the same way, yields a solution which leaves less residue on evapora-



tion—we have an evidence that the specimen under examination is a mixture containing a body more soluble than the bulk ; and if by these treatments we find the residues are uniform in quantity it might be taken as an indication, though not an absolute proof, that the body under examination was a definite compound and not a mixture. Indications of this kind are useful guides in the preliminary examination of bodies the nature and reactions of which have not as yet been determined.

If the point to be ascertained is the extent of solubility of the material in a given solvent, temperature and time become the important points. The material in powder, and in excess of what will dissolve, may be shaken with water (or other solvent) at the required temperature for a considerable time ; then the solution, having been filtered into an evaporating dish and weighed, is to be evaporated to dryness till it ceases to lose weight at  $212^{\circ}$  F. (or other temperature if such be specially indicated). The loss of weight gives the water, and the dry residue indicates the weight of the substance soluble in that quantity of water at the temperature at which the solution was effected. As some substances dissolve very slowly, it is necessary, if the habit of a substance be unknown, to obtain two consecutive results agreeing with one another ; without this the solubility may become so uncertain an indication as to be totally useless—for example, arsenious acid was found by Gmelin to be soluble to the extent of one part in fifty of water if a considerable excess of the acid was used, but one part of the acid was not completely dissolved by 100 of water in eighteen years when the water was used in these proportions. On the other hand, if an elevated temperature be used, which in most cases speedily secures the solution of more of the substance than can be retained when cooling has taken place, the phenomenon of super-saturation becomes a source of probable error, though not usually a troublesome one. A warm saturated solution of alum will become and continue supersaturated for some time after it has cooled, and to insure the solution being normal it requires to be agitated in contact with powdered alum for a considerable time at a constant temperature if accurate results are

desired. In these and similar cases solubility is not a convenient indication of purity, and in those instances where solubility is a convenient and useful indication the chance of errors resulting from the above causes, though much smaller, must not be entirely overlooked.

No definite degree of solubility can be laid down as pertaining to colloid bodies such as gum acacia or tannic acid when treated with water, as they are soluble in all proportions up to that at which the solutions become so thick as to make it doubtful whether the result is a fluid, a semi-fluid, or a soft solid ; yet the testing of complete solubility is a ready and useful examination as indicating the absence of starch or sand in the gum, or resinous bodies in the tannic acid.

When solvents other than water are to be used the risks of error are multiplied. Acids, alkalies, alcohol, spirit, ether, chloroform, benzene, &c., are those most frequently applied.

Lead and barium precipitates, spoken of as soluble in nitric or hydrochloric acids, will give false indications if the acids are used in the concentrated state ; on the other hand, a very dilute solution of copper sulphate may fail to give a precipitate on the addition of ammonia, and a saturated solution of the same if treated with strong solution of ammonia gives a copious precipitate which does not redissolve on the addition of a considerable excess of the ammonia unless diluted with water.

The solvents of organic origin are never obtainable of absolute purity ; the amount of impurity is variable, and with this a variable degree of solvent power results ; nevertheless, when used with judgment, useful indications may be conveniently obtained in a variety of cases. When solubility in 'spirit' is spoken of, rectified spirit (56 O.P.) may be understood, unless 'weak spirit' is the term used, in which case proof or under should be understood. When 'alcohol' is directed, absolute alcohol of the British Pharmacopœia should be used. The expression 'weak alcohol,' though incorrect, is sometimes met with, and may be understood as alcohol and water in which water is more than half. Benzol and benzene are names in common use in England as applied to the volatile distillate from coal-tar,

boiling at about  $180^{\circ}$  F., and of gravity about 800. In America we find the term 'benzin' restricted to the low-boiling distillate from the native petroleum: this must not be confounded with the former, as their solvent powers, though generally similar, have some marked differences; in English works these latter are spoken of as petroleum spirit or petroleum ether.

When ether and chloroform are spoken of as solvents, the commercial qualities are sufficiently pure unless further precautions are specially indicated.

Of recent years the process of 'shaking out' has grown into considerable importance, and is often conveniently applicable to pharmaceutical testing. A few words upon its method of application may be useful in this place. The essential feature in the operation is the selection of two solvents which, after mixing, will separate again. The nature of these solvents must be such that when shaken in the presence of an impure body more or less soluble in each, the matters present will become distributed between the two solvents in something like the ratio of their solvent powers. By a judicious use of separable solvents alkaloids may be separated from the mucilaginous, resinous, and oily bodies with which they are associated in the plants which yield them, and this is greatly facilitated by rendering the condition of the solution alternately acid and alkaline.

Suppose, for example, a particular vegetable, the composition and behaviour of which were already known, and that water, sulphuric acid, ammonia, and chloroform were the appropriate reagents for its treatment: the material first treated with water and acid yields besides its alkaloids much mucilaginous matter and vegetable acids, together with smaller proportions of colour, resin, oil, and wax; the solution in its acid condition being shaken with chloroform vigorously for a few minutes and allowed to settle, the chloroform will contain the bulk of those bodies more soluble in it than in water, such as the wax, oil, resin, and probably some of the colouring matter and acids, but not the alkaloids, which by union with the sulphuric acid are freely soluble in water, but not in the chloroform—this latter may contain a trace, but probably a trace only, of the



alkaloids, and it will probably leave in the watery solution traces of those bodies which it is intended to extract, and when the behaviour of the material has not been previously ascertained by experiment it may be a question whether the acid solution should be again shaken with a fresh portion of chloroform, and separated before further treatment. After the acid solution has been thus purified, it may be rendered alkaline with ammonia, this throwing the alkaloids out of combination, rendering them freely soluble in chloroform and sparingly soluble in water, but not materially affecting the solubility of the other constituents in the two solvents used ; consequently, on again shaking with chloroform, the alkaloids pass into the chloroform with very little contamination of other matters, and after a careful separation and evaporation they may be weighed, giving a close approximation to the quantity of pure alkaloids (mixed if there be several) contained in the material operated upon. The alkaline liquor after separation from the chloroform will contain traces of alkaloids, frequently so small as to be of no practical importance ; but if this point has not been settled by previous experience a second agitation with chloroform should be had recourse to, with the view of insuring the sufficient extraction of the alkaloids from the ammoniacal liquor, and the second portion of chloroform should be evaporated separately and its yield noted as a guide in subsequent operations upon the same substance. The alkaloid obtained by this process is usually pure enough to be a guide as to the quality of a drug or the standardisation of a preparation ; but, if a higher degree of purity be desired, the dissolving of the alkaloidal residue with a small quantity of acidulated water will generally leave behind a trace of fatty matter, &c., and a repetition of the acid and alkaline shakings will further purify it at the expense of a trifling loss.

One point in this process which is apt to give trouble is the disposition which many vegetable infusions show to emulsify the solvents and not give a sharp line of separation. This tendency is increased by the presence of insoluble particles of suspended matter, therefore the watery solution should be



rendered bright by filtration if that can be accomplished without loss of alkaloid, and without inconvenient dilution. It may often be done by filtration through a ball of absorbent cotton, about  $\frac{1}{6}$  of an inch in diameter, pressed loosely half-way down the tube of a small glass funnel. A few drops of water passed through after the filtration suffice to wash out the absorbed infusion, and the liquor is made clear with an insignificant addition to its bulk and practically no loss of alkaloid.

The most convenient separator for this 'shaking out' process is a pear-shaped globe of two or three ounces capacity, with a stoppered neck at the top, and a tube of two or three inches at the bottom, having a glass tap. The aqueous liquor and the separable solvent after being shaken together in this vessel are allowed to stand till the fluids are again satisfactorily separated, when the heavier may be drawn off by the tap, and if this is well performed not a drop of it should remain mixed with the superstratum.



In the typical example just described the aqueous liquor would remain in the separator from the beginning to the end of the process; the impurities having been washed out of the acid liquor, the ammonia is added to the liquor remaining in the separator, and the washing out with chloroform repeated; the risk of loss is minimised; but if ether, benzine, or other solvent lighter than water is used the decantation by means of the neck is not so safe and effectual an operation, and it becomes necessary to run off the aqueous liquor from the tap and return it to the separator for the second part of the treatment.

Spirit cannot be used in connection with water, ether, or chloroform as a separable solvent, but it is possible to use it in connection with oil in a limited number of cases; thus 3ij of almond oil shaken with 3ij of orange-flower water and separated is perfumed with orange-flower, and this in turn shaken with 3ij of strong spirit imparts much of its odour to the spirit; but in this case the action is much too imperfect to have any analytical value, and the very imperfect removal of the odour

from the water by agitation with oil indicates that the distribution between the two solvents is not strictly proportionate to their solvent powers.

### Detection of fixed impurity in a volatile liquid.—

In many instances the B.P. directs that a liquid under examination should evaporate without fixed residue, at other times the expression used is 'with little or no fixed residue.' The latter is the more reasonable requirement, as in the majority of cases the absolute freedom from fixed residue is only theoretically possible. A convenient mode of operating is to put a single drop of the liquor on a clean watch-glass or slip of glass such as is used for microscopic slides, and put it in a position exposed to a gentle heat without dust. A drop of distilled water treated thus rarely fails to leave a small but quite visible residue. Water has such extensive solvent powers that if it were possible to make it pure it could not be preserved pure in ordinary vessels. Rectified spirit, on the other hand, will generally evaporate without visible residue. The proportion of fixed matter that can be detected in this way is very minute. One drop of water holding in solution  $\frac{1}{1000}$  of a grain of sulphate of lime leaves a very conspicuous residue. A drop containing  $\frac{1}{10000}$  of a grain is still conspicuously impure. When the quantity is reduced to  $\frac{1}{100000}$  it still shows more residue than distilled water from which it was prepared, but not a very conspicuous difference. In a parallel experiment in which the latter dilutions were made with rectified spirit the  $\frac{1}{100000}$  of a grain contained in one minim of spirit left a residue quite conspicuous when viewed by reflected light, but not so readily seen on looking through the glass. We may thus conclude that on a drop of any liquid being evaporated on clean glass and leaving a residue not readily visible the weight of the fixed impurity must be insignificant, unless the residue be of a varnish-like character, in which case it usually becomes visible if the finger be lightly drawn over the surface. When this mode of examination is in use it is advantageous to obtain two or three small residues such as those noted above, to enable

a better judgment to be formed when a similar residue is obtained in actual testing.

**Estimation of fixed ash.**—In various cases the B.P. requires that chemicals either volatilise without residue or burn off without ash. For this purpose a piece of clean, bright platinum foil heated over a spirit lamp or Bunsen flame is the best appliance. Glass is out of the question, and porcelain does not show a white residue so well. If the operation be performed critically, the total absence of residue is only to be expected when the material under examination is a product of sublimation. We may expect carbonate or chloride of ammonium to volatilise without a residue, but not nitrate. We may expect benzoic acid to be free from fixed residue, but not to find citric acid to be free from ash. If we take cognisance of  $\frac{1}{10000}$  of a grain of residue, which is a very visible quantity if on a slip of clean glass, we cannot expect a grain of citric acid to burn off and leave less than this. In some cases the requirement is more cautiously expressed : for example, tartaric acid is said to leave *no residue, or only a mere trace*. In burning off any substance which is not a product of sublimation, we may be satisfied if one grain leaves an ash which we judge, by comparison with a known standard, to be not more than  $\frac{1}{10000}$  of a grain. Under the characters and tests of cotton-wool we find the British Pharmacopœia allows a very liberal latitude for ash, that is that it shall not contain 1 per cent. It may easily be purified to contain less than that, though I have not been able to obtain it free from the last traces, even after long-continued maceration in hydrochloric, hydrofluoric acids, &c. The requirement that hydrochlorate of morphia and sulphate of quinine should burn away without leaving any residue, if understood literally, would condemn many good samples.

**Colour estimation.**—In some cases the quality of a drug may be estimated by the colour it imparts to solvents. A variety of instruments have been devised under the names of chromometers and tintometers for facilitating the comparison



of colours with standards ; but these, though valuable to dyers and some others, scarcely require notice now, beyond the general remark that coloured solutions are best compared when diluted to pale tints. Square flint bottles are convenient vessels for the observation when it is not desired to go to the trouble of setting up a special piece of apparatus for a more critical examination. When a little more exactitude is required, a convenient arrangement consists of a pair of small beakers resting by their rims in circular holes cut in the lid of a card-box, with corresponding holes in the bottom of the box, the whole being supported a few inches above a sheet of white paper, upon which a good light falls. In this arrangement two inches' depth of the solution to be examined may be put into one beaker, and the standard solution is poured into the other beaker, till, on looking down through the solutions to the white paper, the tints appear of equal depth. The value of the drug is judged normal if the depth of the standard solution is found to be two inches, and its quality would be judged as above or below what is considered its normal if the depth of the standard solution exceeds or falls short of the two inches of liquor under examination.

**Colour reactions.**—In using litmus, &c., the reaction is more delicate with the tincture than with the paper, and for any special purpose a number of small drops of the tincture upon a white slab readily gives delicate indications.

In testing the oxidation of iron solutions with bichromate of potassium a white slab spotted with small drops of solution of ferricyanide of potassium is the most convenient arrangement. In testing the neutral point of solution of acetate of ammonium (which see) with acetate of lead, a polished black surface spotted with neutral acetate of lead solution shows the reaction most clearly. I have been in the habit of using two plates of glass cemented together with black pitch for this purpose, but a flat dispensing bottle filled with any dark liquid answers very well and is always ready to the hand of the dispenser.



**Precipitates.**—When the weight of a precipitate is to be ascertained there are sundry points which require to be carefully noted : first, that the reagent has been added in such proportion as to throw down all the precipitable matter ; then, that the time allowed and temperature are suitable, some precipitates going down slowly, some are expedited by boiling, others by reduction of temperature. Precipitates are usually best collected and washed upon small, circular, white filtering papers, and if the precipitate is to be weighed upon the filter, which is usually the case with organic precipitates, two of these papers of equal size should be used, one cone being placed within the other, the precipitate being collected on the inner cone. All the washings, &c., being passed through both papers, they are subject to the same solvent actions, friction, &c., and when the operations are complete the outer cone of paper is taken as a counterpoise to that which contains the precipitate. The washing of the precipitate should be continued with the appropriate fluid (hot water, cold water, spirit, ether, &c., as the case may be) till a drop evaporated on a clean slip of glass leaves a scarcely visible residue, regard always being had to the degree of solubility of the precipitate itself. For example, quinine, morphia, &c., though sparingly soluble, cannot be washed without some loss, and the extent of loss becomes important if the washing be unnecessarily prolonged. Of course the washings never cease to leave a residue, however long they may be continued. It is impossible to make a satisfactory allowance for the quantity of the precipitate that may be dissolved by the washing water, as this quantity is very uncertain, the washings usually being far from saturated. In any case, where greater exactness is aimed at and long-continued washing appears important, the precipitate may be washed with a saturated solution of the same substance. Thus, if morphia were long washed with water a loss would be involved, but if it were washed with a saturated aqueous solution of pure morphia the process might be continued any length of time that might be necessary for the removal of the precipitant or other adhering matters, which would still be freely extracted by the water which was

saturated with pure morphia, and consequently incapable of causing loss of alkaloid. In most cases where a precipitate is to be weighed upon the paper the whole should be dried at about  $212^{\circ}$  F. till it ceases to lose weight, and then should be cooled and weighed with as little exposure to the air as possible. All dry substances condense some moisture if exposed to the air; some, without being deliquescent, will absorb a considerable amount.

Inorganic precipitates are usually ignited before being weighed, the paper filter upon which they have been washed being reduced to ashes, and the ash weighed along with the precipitate—the weight of the ash being estimated and deducted in cases where fine results are aimed at, or neglected where a rougher estimate only is required. In all cases it is essential that the whole of the carbonaceous matter should be burned off, and the facility with which this is effected depends much upon the precipitate and filter being well freed from soluble matters and thoroughly dried. The incineration is usually effected in a platinum or porcelain capsule heated over a Bunsen burner, the filter being inverted into the capsule and the precipitate shaken loose. The paper burns more freely if it lies lightly on the surface, but care is required lest a current of air should carry away the light ash when burned. When all the carbon is consumed, which is judged by the whiteness of the ash if the precipitate itself be a white one, the precipitate may be weighed, but if the precipitate consist of oxide of iron or other dark material the safest plan is to repeat the burning and weighing till two consecutive weighings are identical. Care is required in this case as in the former to avoid exposure to the air in cooling, that the absorption of moisture may not take place. The gross weight of capsule, cover, and precipitate having been ascertained, the weight of the capsule and its cover, which ought to be previously known, should again be checked to prevent the chances of error consequent upon their wear or fracture.

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## ACACIÆ GUMMI

Gum acacia in times of scarcity is often mixed with qualities which are imperfectly soluble. In the unground condition these are sufficiently evident by their hardness and glassy fracture. Powdered gum is more liable to sophistication either with those varieties which form only a glairy mucilage or, less probably, with dextrine. The latter is readily detected by its taste; the absence of the former can be judged by the gum forming with its own weight of water a mucilage which will pour in a thin smooth stream, slightly breaking into drops as it falls. The imperfectly soluble gum makes a mucilage which pours in an irregular stream, somewhat resembling white of egg, while the mucilage of good gum more resembles treacle in its flow. The presence of starch is guarded against by the pharmacopœial requirement that iodine shall not give with it a blue coloration.

## ACETUM

Vinegar holds so unimportant a place in pharmacy as scarcely to demand the examination which the pharmacopœia denotes. The only common sophistication is the substitution (or mixture) of acetic acid obtained by the destructive distillation of wood for that produced by the acetous fermentation of a weak alcoholic liquor. This, though not to be justified, cannot be said to have any therapeutic importance, and can best be judged by a comparison of the flavour with a standard sample. It is not within our present scope to consider the circumstantial evidence which might indicate the process by which any sample has been made. Malt vinegar usually contains a little sulphuric acid—if this be not in excess, a fluid ounce of the vinegar precipitated with one grain of barium chloride and filtered will not give any further precipitate with more of the barium salt. The absence of copper, lead, and iron should be indicated by the vinegar after neutralisation with ammonia, giving no black coloration with sulphydrate of ammonium. A fluid ounce of



good ordinary vinegar will dissolve 18 grains of carbonate of magnesium, but not an additional grain. The B.P. standard would theoretically require the solution of 19 grains. With a weak acid, such as vinegar, solution takes place very slowly towards the end of the action, and perfect solution is somewhat obscured by the colouring matter, but the process is quite accurate enough for the article, and a gentle heat saves time. The numbers commercially applied to vinegars as '18, 20, or 24 vinegar' are said to have originally denoted the number of pence per gallon charged by the maker, and, though they have lost that meaning, they are still in use as indicating the grades of quality.

ACIDUM ACETICUM

This acid should contain 33 per cent. of the true acid,  $\text{H.C}_2\text{H}_3\text{O}_2$ , in which case 182 grains will dissolve 47 grains of carbonate of magnesium, but not 48 grains. If it answers this test and leaves no fixed residue, the only probable impurity is sulphurous acid, which according to B.P. is to be sought for by adding f3i of hydrochloric acid and 3i of water to f3i of the acid and 3i of zinc in a flask, when, if  $\text{SO}_2$  be present, the nascent hydrogen converts it into water and  $\text{H}_2\text{S}$ , which may be detected by placing in the neck of the flask a slip of paper moistened with solution of lead acetate, when gradual blackening of the paper will take place from the formation of lead sulphide.

A much readier test for this impurity is to add a drop of tincture of iodine to a drachm of the acid, which gives it a yellow-brown tint if the acid be pure, but is instantly decolorised if there be sulphurous acid present to the extent of  $\frac{1}{160}$  of a grain in the fluid drachm.

The B.P. also requires that the acid should give no indication of copper or lead when treated with sulphuretted hydrogen. It is, however, more convenient to neutralise the acid with ammonia and test with sulphhydrate of ammonium, when the coloration may indicate copper, lead, or iron.



The absence of a precipitate with silver nitrate should show its freedom from HCl, and freedom from precipitation with barium chloride indicates the absence of sulphates. The presence of a trace of calcium phosphate may sometimes be detected, resulting from an imperfectly purified animal charcoal having been used to decolorise the acid. It is not a common impurity, and would be indicated by a faint cloudy precipitate appearing when the acid is treated with excess of ammonia or its carbonate.

### ACIDUM ACETICUM DILUTUM

This should stand the tests for impurities which are indicated under acetic acid, and in the estimation of its strength by the carbonate of magnesia a fluid ounce should dissolve 15 but not 16 grains.

### ACIDUM ACETICUM GLACIALE

This should stand the same tests for impurities as are described under acetic acid. The strength of the glacial acid is usually judged by its retaining its glacial condition until the temperature rises to 60° F., though it may remain fluid after the temperature has fallen considerably below that point.

Its strength may also be estimated by carbonate of magnesia : a fluid drachm of the acid in half an ounce of water should dissolve 45 grains of the magnesium carbonate, but not 47 grains; this test is, however, unnecessary when the freezing and melting points have been found correct.

### ACIDUM ARSENIOSUM

The crude white arsenic of commerce is often nearly pure, but is not dependably so unless obtained in clean cakes or recrystallised for pharmaceutical use. The crude white arsenic may contain metallic arsenium, sulphur, and antimony; the presence of these need not be feared either in the clean cake arsenic, whether transparent or opaque, or in that supplied for

pharmaceutical use as crystalline arsenious acid. The B.P. estimates the quantity of arsenious acid present by the quantity of iodine required to convert it into arsenic acid when treated with bicarbonate of sodium and water. For this operation 4 grains of the arsenious acid with 20 grains of sodium bicarbonate boiled in an ounce of water should promptly dissolve, without a yellow coloration, 10 gr. of iodine, but should become coloured on the addition of half a grain more of the iodine.

The theoretical quantity is 10.26 of iodine to 4 of arsenious acid, and this test would limit the error on either side to 0.1 of a grain in the arsenious acid. As the B.P. directs the examination with volumetric solutions and apparatus, a greater degree of accuracy might be obtained if the pharmacist had facility for weighing the required 4 grains with greater accuracy than to the tenth of a grain. If a narrower limitation were desired, it could be obtained with equal facility by operating upon 8 or 16 grains of the acid and 20 or 40 of iodine.

In practice I have found the commercial powdered arsenic to decolorise a little more than the above quantity of iodine, probably from the presence of some suboxide.

This test is quite unnecessary with clean white arsenic in the sublimed lumps which commonly appear in commerce.

### ACIDUM BENZOICUM

For medicinal use benzoic acid is preferred which is not chemically pure, a contamination of the aromatic oil of benzoin being regarded as advantageous. The aromatic odour is apt to give the impression that the acid volatilises at common temperatures, but after long exposure to the air, when the aromatic odour has passed off, there is but little diminution of weight. The only adulteration to which the official acid is liable is the admixture of pure benzoic acid obtained from the gum by the treatment with lime, &c., or with benzoic acid artificially produced: such deviation from the official requirements cannot be detected by chemical means, but may be judged by deficiency of the aromatic odour. The U.S.P. also tests for cinnamic acid, a not improbable impurity, but an unimportant one. It

would have been more philosophical on the part of the compilers of the B.P. to have used the old name 'Flowers of Benzoin,' since they require it to be produced by sublimation from the gum, and that it shall contain at least some of the impurities naturally present when thus obtained.

### ACIDUM BORICUM

The acid of commerce rarely contains any impurity of consequence, sulphuric acid and soda being the most common. The latter is detected by exposing a little of the boric acid on a loop of platinum wire to the flame of a spirit lamp, when any considerable yellow colour in the outer part of the flame would indicate soda. A faint yellowness is almost invariably produced, but that is not sufficient to condemn the sample, as a very small trace of soda will produce this reaction, and the colour will be increased by moistening the acid with a drop of hydrochloric acid, as this assists the volatilisation of the sodium as chloride.

A solution of 15 grains of ordinary boric acid in 1 ounce of water is almost certain to produce a slight milky appearance on the addition of barium chloride, but this may pass if it does not exceed the milkiness produced by adding a drop of dilute sulphuric acid to 4 ounces of distilled water containing a grain of chloride of barium. The absence of coloration on the addition of sulphhydrate of ammonium should indicate its freedom from iron, copper, and lead.

### ACIDUM CARBOLICUM

Samples of carbolic acid which are crystalline, either white or pale pink, and without any strong tarry odour, may be accepted as suitable for medicinal use. The B.P. is rather indefinite in the degree of purity required, and the pharmacist would naturally aim at the highest purity in acid for internal use, while the less pure might be used for external applications. The official requirement is that one part of the crystals shall dissolve in 12 to 18 parts of water, and the solution should be clear and colourless, or nearly so ; any insoluble brown matter separating as dark oily drops should not have more than a faint

tarry odour. The U.S.P. is more explicit in this direction : it says that one volume of the liquid hydrated acid with one volume of glycerine should make a clear solution not rendered turbid by the addition of three volumes of water ; turbidity of this mixture would indicate the presence of cresylic acid or the less soluble tar oils.

In the inferior qualities sulphur compounds are often present, and perceptible by their odour, though the odour sometimes suggests the presence of  $H_2S$  when lead paper does not indicate it.

The statements regarding melting point and solubilities will probably yet undergo revision as further improvements take place in the process of manufacture. For the present, it is a fair rule that the more the acid is purified the higher becomes its melting point and the lower its boiling point.

Disinfecting acid of good quality should not be darker than a light brown colour, have but little of the heavy tar odour, and be soluble to the extent of f 3i in f 3iii of hot water, depositing a little as it cools. Or f 3i should dissolve with little or no residue in f 3x of solution of potash.

### ACIDUM CHROMICUM

Chromic acid is always contaminated with a little sulphuric acid and moisture adhering to the surface of the crystals ; these do not materially affect its medical uses. Chromate of barium, though almost insoluble in water, is freely soluble in hydrochloric acid ; hence the faint opalescence produced when barium chloride is added to a solution of one or two grains of chromic acid in an ounce of water indicates the presence of the sulphuric acid which the process of production has left adhering to the crystals ; but on the addition of ammonia a free precipitation of barium chromate takes place.

### ACIDUM CITRICUM

The probable impurities in citric acid are traces of lead, copper, or iron, sulphuric, tartaric, or oxalic acid, and organic colouring matter.



If an aqueous solution neutralised with ammonia be not darkened by sulphhydrate of ammonium, the absence of lead, copper, and iron will be indicated. A little of the acid added to clear lime-water would render it turbid if oxalic acid were present. Freedom from tartaric acid is indicated by the absence of a precipitate when 5ss of citric acid dissolved in 3i of water is added to a solution of 5ss of potassium acetate dissolved in 3i of water.

Twenty-three grains of citric acid in an ounce of hot water should dissolve 15 grains of carbonate of magnesia, but not an additional grain.

The acid should burn away without leaving more than a barely visible trace of ash, indicating the absence of alkalies and earths, which would not be detected by the previous tests.

### ACIDUM GALLICUM

This acid is commercially nearly pure, a little colouring matter or a trace of tannin being the most probable impurities. A drachm of the acid should make a clear solution with half an ounce of water at 212° F. On cooling to 60° F. it becomes a magma of crystals; when broken up and thrown into a funnel plugged with absorbent cotton, and water dropped upon it till f 3i of clear solution has percolated, this percolate on evaporation leaves not more than 1 grain of dry residue. The theoretical weight of the residue should be 0.54 grain, but if the temperature be lower and the acid pure the weight may be less than half a grain. This test will exclude any notable contamination with tannin and the great majority of other impurities which it might contain. Tannin may be definitely identified in any excessive residue by the B.P. test, a solution of gelatine being precipitated by tannic but not by gallic acid. It is, however, unnecessary to apply this latter test if the former gives satisfactory indications.

### ACIDUM HYDROBROMICUM DILUTUM

Hydrobromic acid made by the official process might have traces of either free bromine or sulphuretted hydrogen, but if

these are not evident by their odour they need not be further sought for. Sulphuric acid is a more probable impurity, and should be tested for with chloride of barium. Acid made by the old process—decomposing KBr with tartaric acid—was a very impure product, containing, besides free hydrobromic acid, potassium bromide, acid tartrate of potassium, and free tartaric acid, and would be distinguished from the official acid by its leaving a considerable fixed residue on evaporation, the B.P. acid leaving only a trace. A fluid ounce of the official acid should form a clear solution with 27 grains of carbonate of magnesium, but not with 29.

### ACIDUM HYDROCHLORICUM

The impurities most commonly found in hydrochloric acid are sulphuric acid, free chlorine or nitrous compounds, iron and arsenium ; lead, copper, and the earthy and alkaline metals are also possible impurities, though scarcely demanding our special testing. If a drop of the acid evaporated on a clean slip of glass leaves a scarcely visible mark, the latter impurities need not be sought for. Arsenium chloride, being freely volatile, would not be indicated, and must be sought for by boiling a piece of clean copper in the acid diluted with four volumes of water ; a small trace of arsenium would impart a steel-grey colour to the copper. The presence of iron, lead, or copper in the acid would be detected by adding ammonia in excess, and then a drop of sulphydrate of ammonium, which would cause no coloration if the acid were pure, but a blackening in the presence of any of these metals. Any considerable contamination with iron would be visible as a brown precipitate before the addition of the sulphydrate, and any considerable quantity of copper would be seen in the blueness of the ammoniacal solution ; but the precipitation as sulphides is so much more delicate that it will often give indications where pure ammonia fails to do so. A drop of solution of barium chloride should not cause any milkiness in the diluted acid ; an immediate appearance of a precipitate would indicate the presence of sulphuric acid in quantity sufficient to disqualify the acid for pharmaceu-

tical use, and the appearance of a trace of precipitate only after a lapse of several minutes would indicate so small a trace as would not render it unfit for use in pharmacy, but would prohibit its application to analytical purposes. The absence of free chlorine or nitrous compounds is to be indicated, according to B.P., by the blue colour of indigo not being bleached. To detect a small trace of free Cl, the quantity of indigo solution should be only sufficient to give a pale blue tint to two or three drachms of water, and then an equal volume of the strong acid poured in ; the colour should be unchanged after the lapse of several minutes. Litmus, cochineal, and many other organic colours are also readily bleached by chlorine, but indigo gives the clearest results, especially with the nitrous impurities. The U.S.P. directs chlorine to be sought for by the addition of iodide of potassium to the diluted acid, when the absence of iodine coloration would indicate that no free chlorine was present. If to a pure hydrochloric acid a small trace of chlorinated lime be added, the free chlorine is strongly indicated by its liberating iodine from the iodide ; but if instead of chlorinated lime we add a drop of nitric acid the action is much less marked, and, as this better represents the probable impurity in a commercial acid, the indigo test is to be preferred.

Among the B.P. tests is included one for sulphurous acid ; the hydrochloric acid, being diluted with four volumes of water, is made to act upon zinc, when, if sulphurous acid be present, it is decomposed and evolved as  $\text{H}_2\text{S}$ , the presence of which is indicated by a strip of paper moistened with solution of lead acetate and suspended in the neck of the flask in which the operation is performed. Sulphurous acid is an impurity which I have not met with in the hydrochloric acid of trade, but if present would be much more readily detected by its decolorising action upon permanganate of potash or solution of iodine. Nothing can be more convenient to the pharmacist than to add a single drop of the B.P. tincture of iodine to a drachm or two of the acid, and if it contains  $\frac{1}{200}$ th of a grain of  $\text{SO}_2$  it will be immediately deprived of its yellow-brown colour ; but in the absence of  $\text{SO}_2$  the iodine colour will be permanent and conspicuous in even



three or four times this quantity of acid. Of course it should be remembered that the decolorisation of the iodine is not absolute proof of the presence of  $\text{SO}_2$ , but it proves the presence of some reducing agent, and the permanence of the iodine colour is positive evidence of the absence of  $\text{SO}_2$ .

The strength of the acid is sufficiently indicated by its density being 1.160, or by f3i with 3ss water making a clear solution with 26 grains of carbonate of magnesium, but not dissolving 2 grains more.

#### ACIDUM HYDROCHLORICUM DILUTUM

This should of course stand the same tests as the former, but its gravity should be 1.052, and the solvent power of f3ij should equal 23 grains of carbonate of magnesium, but not 25. Theoretically, f3vi = mag. carb. 47.5.)

#### ACIDUM HYDROCYANICUM DILUTUM

The only respect in which this acid requires examination is as regards its strength. This is most readily performed by mixing f3ij of the acid with f3ij solution of potash and adding  $6\frac{1}{2}$  grains of nitrate of silver; this should produce a precipitate dissolving as it forms; the precipitate ceasing to redissolve before all the nitrate of silver is dissolved would indicate that the acid was deficient in strength, and the extent of deficiency might be approximately determined by the quantity of acid necessary to be added before the solution of the precipitate could be effected.<sup>1</sup> The B.P. directs the volumetric estimation of hydrocyanic acid to be 'performed speedily so as to prevent loss by volatilisation,' a precaution which is not at all necessary; the acid, having been combined with an excess of caustic alkali at the first stage of the process, is no longer liable to evaporate. Two examinations of the same acid, one performed speedily

<sup>1</sup> See further, in Proctor's *Pharmacy*, supplementary notes on pharmaceutical testing.



and the other purposely exposed to the air for half an hour after the addition of the alkali, gave me identical results.

The Prussian blue test as given in the B.P. is simply intended to identify the hydrocyanic acid, which, when mixed with the oxides of iron and caustic potash, is converted into ferrocyanide of potassium, and then, when the mixture is acidulated with HCl, the iron being in excess, the blue ferrocyanide of iron is formed.

### ACIDUM LACTICUM

The mineral acids, the heavy metals, sugar, and glycerine are the chief foreign matters to be guarded against in lactic acid.

Sulphuric and hydrochloric acids are to be sought for, as usual, with barium chloride and silver nitrate, and the metals by adding ammonia in excess to a portion of the acid diluted with 10 volumes of water, and then a few drops of sulphhydrate of ammonium; a white precipitate would indicate zinc—a probable impurity introduced in the process of manufacture; a dark-coloured precipitate would possibly also contain zinc, along with iron, copper, or lead. In either case the acid is not sufficiently pure for medicinal use.

Sugar, if present in lactic acid, would be of such varieties as reduce copper sulphate to the suboxide in the presence of alkali. To fʒi of lactic acid in fʒi of water add 1 grain of copper sulphate, 4 or 5 grains of Rochelle salt, and sufficient solution of potash to give a strongly alkaline reaction; then bring the whole to the boiling point, and if sugar be present the colour first changes to yellow and then red, the suboxide of copper being precipitated.

The U.S.P. directs glycerine to be sought for by converting the lactic acid into lactate of zinc, and extracting with absolute alcohol, which will dissolve glycerine if any be present, and on subsequent evaporation will leave it as a sweet residue; many of the lactates are soluble in spirit, hence the necessity for using oxide of zinc rather than soda, potash, or lime in this test.

Lactic acid does not satisfactorily act with magnesium carbonate to indicate its strength, nor yet with bicarbonate of soda ; the colour reaction with litmus is not sharp enough in the latter case, even if aided with heat, to get rid of the carbonic acid. The B.P. volumetric method acts satisfactorily, as the soda is in the caustic state, and gives a much sharper indication of the point at which the change from acidity to alkalinity takes place. For dispensing-counter purposes it is well to remember that 995 minims of liquor potassæ (if of correct strength) is equal to 1,000 grain measures of volumetric solution of soda ; consequently, if the liquor potassæ is *a reliable sample*, the lactic acid may be readily tested by taking 11 minims (or 12 grains) of lactic acid, two drops of tincture of litmus, and two drachms of water : this should retain its pink colour on adding 95 minims of liquor potassæ, but becomes blue on adding 5 minims more.

In testing the dilute lactic acid, 73 minims (=70 grains) may be used with the same quantity of alkali.

### ACIDUM NITRICUM

Nitric acid is frequently contaminated with  $\text{N}_2\text{O}_4$ ,  $\text{H}_2\text{SO}_4$ , or  $\text{Cl}$  ; also basic impurities. The U.S.P. adds free iodine, iodic and arsenic acids as impurities to be sought for.

In the absence of a yellow colour  $\text{N}_2\text{O}_4$  need not be feared. In the presence of a yellow colour iron, iodine, or  $\text{N}_2\text{O}_4$  may be suspected—the latter most probably.

If a drop of the acid evaporated on a clean glass leaves a scarcely visible residue, the bases generally may be considered absent ; also arsenic and iodic acids, which are only volatilised at a high temperature with decomposition. Sulphuric acid is to be sought for by diluting the acid with several volumes of water, and adding barium chloride solution sparingly. It must be remembered that barium nitrate is not freely soluble in dilute nitric acid, and almost insoluble in strong. The neglect to dilute the acid freely sometimes leads an inexperienced hand to conclude that sulphuric acid is present where there is none. In testing for chlorine with silver nitrate no special precaution

is required. Iodine need not be sought for in a colourless acid.

Arsenium is not a common impurity in nitric acid ; if it be present in the sulphuric acid used in the process of manufacture, it would be converted by the nascent nitric acid into arsenic acid, which is not volatilised except at a high temperature with decomposition, so that under ordinary circumstances the nitric acid is free from it.

If a drop of nitric acid evaporated on a clean glass leaves a palpable residue, it should not be accepted for pharmaceutical use, but if it still be desired to seek some clue to the nature of the impurities add an excess of ammonium carbonate to a drachm of the acid diluted with 3 or 4 volumes of water : a white precipitate would indicate alumina or lime, a brown one iron, a blue coloration copper ; on subsequent addition of a little sulphhydrate of ammonium a white precipitate would now indicate zinc, a black one probably lead, unless iron or copper had been previously indicated, in which case all three may be present, as well as zinc and some other less probable metals.

A fluid drachm of nitric acid in an ounce of water dissolves 40 grains of magnesium carbonate, but not 42.

### ACIDUM NITRICUM DILUTUM

Three fluid drachms should dissolve 23 grains of magnesium carbonate, but not 25. Other tests are the same as for the strong acid.

### ACIDUM OLEICUM

The impurities to which this is subject are stearic and palmitic acids, rancid fatty matters, and undecomposed oil. For pharmaceutical use only those samples should be accepted which are nearly odourless and tasteless, and of a yellow colour, free from red or brown tint. It should make a clear solution with an equal volume of alcohol, in which case it is satisfactorily free from stearin and margarin ; but the absence of olein is not proved by this test, as this latter is freely soluble in absolute alcohol. The considerable similarity in the solubility of oleic



acid and olive oil makes the solubility a rather inconclusive test, but, as proximate purity is all that is demanded, it may be a satisfaction to note that oleic acid is freely soluble in ordinary rectified spirit, while olive oil makes a milky mixture. Oleic acid makes a milky mixture with proof spirit. Oleic acid containing 20 per cent. of olive oil makes a milky mixture with rectified spirit, but if only 10 per cent. of olive oil be present the solution is clear.

The B.P. directs that oleic acid should make a clear solution with a warm solution of carbonate of potash ; milkiness in this case is also an indication of oil. The presence of other fatty acids is also directed to be sought for by neutralising this alkaline soapy solution with acetic acid, to get rid of any potassium carbonate it may contain, then adding lead acetate, collecting and washing with hot water the precipitated oleate of lead, and then proving its almost entire solubility in ether. Compounds of lead with the fatty acids other than oleic being scarcely soluble in ether, their presence as impurities would thus be detected. The operation, however, requires some care and nicety to secure the absence of carbonate or free oxide of lead in the precipitated oleate, otherwise these would remain insoluble in the ether, and might lead to a false conclusion. The following experiment may be a guide to others. A fluid drachm of oleic acid dissolved in 7 fluid drachms of methylated spirit effervesces on the addition of 10 grains of potassium carbonate previously dissolved in a drachm of water ; when the effervescence has passed off the fluid is clear, and a further small addition of oleic acid does not renew the effervescence nor render the solution milky ; this now, being an alcoholic solution of potash soap in which the fatty acid is in excess, may be precipitated with 20 grains of lead acetate dissolved in a drachm of water and diluted with 7 drachms of methylated spirit : this lead solution should be clear, either by filtration or a *slight* excess of acetic acid, and then poured into the soap solution ; a precipitation of oleate of lead with an excess of oleic acid takes place in the form of an oily fluid sinking to the bottom of the spirit. If this be washed with spirit the excess of oleic acid will be ex-



tracted, and the oleate is reduced to a soft solid which is readily and entirely soluble in two or three volumes of ether.

### ACIDUM OXALICUM

Though oxalic acid is not used in medicine, it is a common chemical in the hands of pharmacists, and if used as a means of estimating alkaline bodies its purity becomes a matter of some importance. As found in commerce it is usually not far removed from purity, and when subjected to a second crystallisation and drying without heat it becomes a very good standard. It should form a clear solution with distilled water, and should burn away leaving an inappreciable ash ; and a solution of the acid treated with excess of calcium carbonate, and filtered, should be converted into almost pure water. Most of the impurities liable to be present in the oxalic acid would be contained in the filtrate, and would be indicated on evaporation. For an examination take 10 grains of the acid in  $\frac{1}{2}$  ounce of distilled water, add to it 20 grains of precipitated chalk which has been washed two or three times with distilled water ; bring it to a boil, and filter through a paper which has been washed with distilled water, and test the filtrate by evaporating a drop upon a clean slip of glass. Operating in this way, I have found with ordinary commercial acid that the quantity of impurity is very small. A drop of distilled water evaporated on glass leaves a visible mark : a drop of the final washing of the chalk left apparently about twice as much and a drop of the filtrate containing the impurities of the oxalic acid left about four times as large a residue as the distilled water.

### ACIDUM PHOSPHORICUM CONCENTRATUM

The most probable impurities in the phosphoric acid of pharmacy are arsenic acid, derived from arsenium in the phosphorus ; phosphorous acid, from imperfect oxidation, or oxides of nitrogen imperfectly removed by the process of manufacture ; and pyro- or meta-phosphoric acids, produced by heat in the

evaporation. Lime, magnesia, ammonia, and sulphuric acid are also impurities which might be present in an acid made from bones without the intermediate step of reducing the phosphorus to its elementary state. The arsenic acid is, according to B.P., to be sought for by the long-continued action of  $H_2S$  and warmth, which gradually precipitates yellow pentasulphide of arsenium. A readier mode is to boil in a test-tube two or three drachms of the acid with a grain of sodium sulphite or a drop of sulphurous acid, continuing the boiling till all sulphurous acid is driven off: by this means the arsenic acid will be reduced to arsenious acid; upon the further addition of a few drops of hydrochloric acid and a little piece of clean copper foil or wire the presence of arsenium would be indicated by the copper assuming a steel-grey colour. If the hydrochloric acid in use has not been previously proved free from arsenium, the risk of an error arising from this cause may be obviated by using, instead, a grain of common salt or chloride of ammonium. The production of an immediate white curdy precipitate on the addition of silver nitrate to phosphoric acid would indicate a chloride, and if a blackening were produced by the same reagent on the application of heat phosphorous acid would be indicated.

Nitrous compounds, if present, are usually sufficiently evident by their odour, but may be further tested for by mixing equal volumes of the phosphoric acid and strong sulphuric acid, and, when cold, dropping in a crystal of sulphate of iron without agitation: the appearance of a dark coloration round the crystal would show the presence of this impurity.

Meta-phosphoric acid is detected, according to B.P., by its power of precipitating albumen in the cold, or, according to U.S.P., by its slowly throwing down a precipitate when the acid is mixed with an equal volume of alcoholic tincture of ferric chloride.

Regarding the less probable impurities, lime and magnesia would be thrown down on the addition of an excess of ammonia to the diluted acid: the lime as a rather gelatinous precipitate, and magnesia as microscopic crystals.

Ammonia which is present in phosphoric acid made by the glacial method would be detected by its odour on the addition of caustic potash or soda in free excess, and sulphuric acid, as usual, by its precipitation with barium chloride; the barium phosphate, though an insoluble salt, is not thrown down in strongly acid solutions. Gmelin also adds iron, copper, and lead to the list of impurities; if the sample of acid to which excess of ammonia has been added is treated with sulphhydrate of ammonium, the absence of a dark precipitate would indicate that none of these three were present.

The strength of phosphoric acid is not readily ascertained by use of carbonate of magnesium as suggested for most of the other acids, but a good approximation may be obtained by dissolving 14 grains of bicarbonate of sodium in half an ounce of water at  $212^{\circ}$  F.; add 50 minims of the phosphoric acid, and bring the solution to the boiling point again; then add other 10 minims of the acid, drop by drop, which should continue to give a slight but visible effervescence up to the last drop.

### ACIDUM SALICYLICUM

If free from colour and the odour of carbolic acid, the only impurity probable in this acid is sodium chloride. From the lightness and bulk of the acid, a little colouring matter may escape notice, but will become more evident if a sample be dissolved in pure spirit and allowed to evaporate to dryness, when any colouring matter becomes visible about the edges of the residue.

Sodium chloride would remain as a fixed residue on heating a little of the acid in a porcelain capsule or on platinum foil.

A further general indication of purity may be obtained thus:—13 grains of the acid, 4 grains of magnesium carbonate, in 3iiss water, on the application of heat should make a clear solution which deposits abundance of crystals on cooling. With 5 grains of magnesium carbonate part of the magnesia may remain undissolved, but no crystals will deposit on cooling. With 6 grains of the carbonate part will remain undissolved on boiling, and no crystals will deposit on cooling.



## ACIDUM SULPHURICUM

The probable impurities are lead, arsenium, and sulphurous acid or nitrous compounds. The two former and the latter are generally present in the cheap commercial acid, but the re-distilled acid as supplied for pharmaceutical use is generally quite satisfactory. Gmelin enumerates the impurities found in common sulphuric acid as  $H_2O$ ,  $HCl$ ,  $NO_x$ ,  $K$ ,  $Pb$ ,  $Se$ ,  $Ca$ ,  $Mg$ ,  $Ti$ ,  $As$ ,  $Zn$ ,  $Sn$ ,  $Fe$ ,  $Cu$ ,  $Hg$ , and  $C$ .

Regarding most of these we need not trouble ourselves, as they do not appear in the acid of pharmacy.

Sulphurous acid or nitrous oxides, if present, are perceptible by their odour ; lead, copper, and iron would be indicated by adding ammonia in slight excess to the diluted acid ; a rusty or olive-green precipitate would indicate iron ; a blue colour, copper ; and a white precipitate, lead ; a small trace of any of these might escape detection in this way, but would give a dark coloration on the further addition of ammonium sulphydrate. Lead usually shows itself as a white precipitate of sulphate of lead on diluting common oil of vitriol with water ; the lead sulphate being less soluble in dilute acid than in strong, and as it is still less soluble in alcoholic liquors, the U.S.P. directs its detection by diluting the strong acid with 4 volumes of alcohol, when any lead present would be thrown down.

Arsenic is to be sought for by boiling a little of the dilute sulphuric acid with a piece of bright copper and a little hydrochloric acid or ammonium chloride, when the arsenic, if present, will give a steel-grey colour to the copper. This is generally more convenient than precipitation of the arsenium by boiling with stannous chloride and strong hydrochloric acid as described in B.P.

The copper test (which is known as Reinsch's) is said to give the steel-grey film when the arsenium forms one part in 300,000 of the solution—a very satisfactory degree of sensitiveness ; the weak point of this test being that when the arsenium is present as arsenic acid the deposit does not take place unless



reduction to arsenious acid is first effected by a reducing agent such as sulphurous acid, and the excess of this must be got rid of by boiling before immersing the copper, or it may mislead by the blackening which is caused by  $\text{SO}_2$  itself.

The stannous chloride (Bedtendorff's test) is said to give indications with so small a proportion as one in a million, but it fails except in the presence of a large excess of free hydrochloric acid.

Selenium is occasionally associated with sulphur in some ores, and, if present in the crude acid, it may escape separation by the process of distillation, as selenious acid is volatile and will be contained in the first portion of the acid distilled. It is precipitated as selenium when the sulphuric acid is boiled with stannous chloride and hydrochloric acid. Arsenium is precipitated by this same treatment, and is the more probable impurity of the two, unless its absence has been proved by boiling with copper as already described, or by other means.

The iron test for nitrous compounds (see Phosphoric Acid) is sometimes applied by pouring a solution of ferrous sulphate carefully upon the surface of a drachm or two of the strong sulphuric acid contained in a test-tube, in which case the blackening caused by this impurity takes place at the point of junction of the two liquids.

If a drop of permanganate solution be diluted with so much water as to have a pale pink colour, and then an equal volume of the dilute acid added, an immediate disappearance of the colour would take place if  $\text{SO}_2$  were present. (See also iodine test for  $\text{SO}_2$  under Hydrochloric Acid.)

The evaporation of a drop of the strong acid on platinum should leave a scarcely visible residue; the alkaline or earthy bases which would not show themselves with  $\text{NH}_3$  and  $\text{H}_2\text{S}$  would be visible as a palpable white residue. The strength of the acid is satisfactorily indicated either by its gravity being 1.843 or by 50 grains in half an ounce of water dissolving 47 grains of magnesium carbonate, or f $\frac{3}{4}$ i of the acid in f $\frac{3}{4}$ i of water dissolving 95 grains of the carbonate.

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ACIDUM SULPHUROSUM,

when prepared by the official process, is not liable to any defect beyond want of strength, and the presence of a little sulphuric acid from oxidation of the sulphurous. When in good condition it should give only a slight precipitate with barium chloride, but this increases with age from absorption of oxygen ; both from this cause and the evaporation of  $\text{SO}_2$  the pharmaceutical acid is often below its legitimate strength. The B.P. requires that it should contain 6.4 per cent. of  $\text{H}_2\text{SO}_3$ , and in that case f 3i diluted with a considerable bulk of water should convert 11.1 grains of iodine into hydriodic acid, which is to be proved by the absence of blue coloration with starch until iodine in excess of this quantity has been added. A simpler operation is to put 11 grains of iodine and 15 grains of iodide of potassium into an ounce vial, pour upon it f 3i of the acid, wash out the measure with a drachm of water, adding this to the contents of the phial, and shake well : the iodine colour should disappear if the acid be of full strength ; this, however, is not often the case, and the degree of deficiency may be roughly estimated by adding successive portions of say 10 drops of the acid till the *brown* colour disappears. There is no necessity for using starch to prove whether or not the iodine has been converted entirely into hydriodic acid, as the colour of free iodine and its disappearance are very sharply marked if the  $\text{SO}_2$  be added by single drops towards the end of the reaction. One hundredth of a grain of free iodine, which is equivalent to  $\frac{1}{18}$ th of a minim of B.P. sulphurous acid, gives a distinct brown-yellow colour to a drachm of solution ; but, though this colour entirely disappears when there is just sufficient  $\text{SO}_2$  to convert the I into HI, a few drops more of the  $\text{SO}_2$  again develops a lemon-yellow colour, removable by a small further addition of iodine or a free dilution with water. This lemon-yellow colour produced by any considerable excess of  $\text{SO}_2$  need not cause any hesitation in the use of this method, as the difference between the lemon and the brown tint is sufficiently distinct,

and the former is accompanied with the odour of free  $\text{SO}_2$ . In the B.P. test the sulphurous acid is diluted with a large bulk of water to prevent evaporation of acid, and no doubt also with the view of ensuring the complete conversion of the sulphurous acid into sulphuric, which under some circumstances does not take place in concentrated solutions. The water for dilution requires to be recently boiled and cooled, otherwise so large a bulk might contain in solution sufficient free oxygen to vitiate the result. Operating in the manner indicated above, the necessity for large dilution is done away with, the boiling of the water is unnecessary, and the colour reaction is quite sufficiently sharp without starch paste.

As a dispensing-counter operation a still more handy method is to operate entirely with B.P. tincture of iodine. Measure  $\text{f}\overline{5}\text{ss}$  of the acid to be tested, pour it into a phial, rinse the measure with  $\text{f}\overline{3}\text{i}$  tincture of iodine, and then add three drachms more of the tincture. In this proportion, half a drachm of the acid to half an ounce of the tincture, the solution should be about colourless, but if still brown add the acid in small measured portions till the colour disappears, and the total quantity used will indicate how much of the sample in hand will have to be used for each  $\text{f}\overline{5}\text{ss}$  prescribed.

Sulphurous acid prepared by other processes may contain other impurities. The evaporation of a drop on glass with a scarcely visible residue, together with the estimation of strength with iodine, are sufficient to establish its satisfactory quality.

### ACIDUM TANNICUM

Tannin of pharmacy is usually nearly pure, and can scarcely be considered in need of special testing. The B.P. statement that it is readily soluble in water (freely soluble would be more correct) sufficiently excludes gallic acid, which is one of the most probable impurities. Tannin is one of those colloid bodies to which we cannot assign any precise limit of solubility. It forms a syrupy solution with its own weight of cold water, and with half its weight of water it forms a pasty mass which is



quite fluid when heated, but stiffens on cooling without depositing any solid particles. The B.P. also adds that it is very sparingly soluble in ether, a statement which general experience contradicts, the tannin dissolving readily in its own weight of commercial absolute ether, either pure or methylated. There are, however, some rather anomalous phenomena in its behaviour with ether, which it is unnecessary to describe here. I only mention the circumstance that it may not be concluded as evidence of the bad quality of any sample of tannin that it is found to be freely soluble in every quality of ether that is attainable. See further in paper on Tannin, its solubilities, etc., at Pharmaceutical Conference, 1889.

The estimation of tannin is a subject upon which much has been written without absolutely satisfactory results, and so far as I am aware no process has been proposed which could be considered suitable for the pharmacist's use. I have, however, made two observations which, while they are not suited to the purposes of the tanner, are of some interest in connection with pharmaceutical tannin.

Fifteen grains of tannin dissolved in half an ounce of spirit and poured into a mixture of a fluid drachm of tincture of perchloride of iron with a fluid drachm of strong solution of acetate of ammonium and two drachms of spirit produces a black magma, which, when well stirred, does not indicate either tannin or iron in solution. This neutrality is best shown by letting a drop of the magma fall upon blotting-paper, when a colourless, or nearly colourless, liquid will spread in the paper beyond the edge of the black drop, and this pale margin may be tested by dropping on the paper, in proximity to it, a drop of tincture of iron on one side and a drop of tannin solution on the other. If the magma contain an excess of tannin, the iron will produce a blackening of the pale margin, but if it contains a deficiency the tannin solution will cause the darkening of the pale margin.

This complete precipitation of tannin by suitable ferric compounds in excess leads us to another mode of examination. Twenty grains of tannin and 20 grains of dry ferric sulphate



dissolved in an ounce of warm distilled water will partially decompose the iron salt ; but if now 40 grains of precipitated barium carbonate be added the complete precipitation of all these chemicals takes place. On heating the mixture to its boiling-point, to expedite the completion of the changes and expel carbonic acid, and then throwing it upon a filter, if the chemicals were pure, pure water would come through. If the tannin should contain matters which do not form insoluble compounds with ferric oxide, these would be found in the filtrate. Examined in this way, I have found tannin of pharmacy nearly pure. Unless there be reason to the contrary, an examination of this kind is not called for.

The solubility of tannin in absolute alcohol is very considerable, 100 grains of alcohol dissolving 160 grains of tannin, forming a thick syrupy solution.

In benzine it only dissolves to the extent of about one part in 60,000.

In chloroform the solubility is slightly greater, possibly in consequence of the small addition of alcohol to the chloroform, which is made with the view of increasing its stability.

## ACIDUM TARTARICUM

The commonest impurities in tartaric acid are moisture and organic colouring matter, which are sufficiently indicated by its appearance. Traces of potash or lime are not uncommon, but usually in too small proportion to be of any importance ; their absence should be indicated by the scarcely palpable ash which remains after exposure to a red heat on platinum foil or in a porcelain capsule. A small but visible ash would not suffice to condemn the sample if it (the ash) consisted of potash or lime, but it might contain also lead, copper, or iron, the first of which is one of the most objectionable impurities and not a rare one, though commonly only in very small quantity. The absence of these metals may be sufficiently proved by dissolving 3i of tartaric acid in 3ss solution of ammonia (.959) and adding

a drop of sulphhydrate of ammonium, when a darkening of the solution will take place if one or more of the three be present. If the precipitate thus formed were sufficiently large to be separated by filtration, the paper filter, after washing with a little water, would turn a buff colour if iron were the only impurity, but would retain its grey or black colour if lead or copper were present. Commercial tartaric acid commonly contains traces of both lead and iron, the quantity of which may be judged by a comparison of the colour yielded with the colour produced in very dilute lead solution of known strength. For example, a drachm of one commercial sample tested with ammonia and sulphhydrate of ammonium gave a colour which was imitated by adding drop by drop, up to 32 minims, a dilute lead solution made by adding one minim of liq. plumbi to an ounce of water ; and the colour of the tartaric solution was reduced to about one-half by acidulating it with HCl. The permanent blackness representing  $\frac{16}{480}$  or  $\frac{1}{30}$  minim liq. plumbi =  $\frac{1}{100}$  gr. Pb in the drachm of acid, and the tint removable by acid, indicated a like quantity of iron.

The official test with potassium acetate is intended to distinguish the tartaric from other vegetable acids by the production of a precipitate of potassium acid tartrate. Oxalic acid is an occasional impurity, and is indicated by a white precipitate being formed on the addition of a solution of calcium sulphate. This reagent is used in preference to other more soluble calcium salts, because it insures the solution being so dilute as not to throw down calcium tartrate, which is a sparingly soluble salt. Lime-water may be used with equal advantage, and is sure to be on the pharmacist's shelves. If the tartaric acid be free from oxalic it may contain lime, which would be indicated by its solution giving a white precipitate on the addition of oxalate of ammonia. Twenty-five grains of tartaric acid in 1 ounce of hot water dissolves 16 grains of magnesium carbonate, but not 17 grains.

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## ALKALOIDS AND THEIR SALTS

A very convenient and useful mode of examining alkaloids and their salts is founded upon the alkaloids in the free state being generally sparingly soluble in water, but freely soluble in ether, chloroform, or benzol, and the sulphates or some other salts of the alkaloids being generally freely soluble in acidulated water, but almost insoluble in ether, chloroform, or benzol. Suppose, for example, that sulphate of quinine were impure from the presence of sugar, gum, and common salt: 3ss of the sample dissolved in 3ss of water with the aid of 3ss of dilute sulphuric acid, the solution shaken with an equal volume of ether (which is the appropriate solvent of quinine, though not a solvent of all alkaloids), would yield nothing appreciable to the ether, which on subsidence and decantation should leave no residue on evaporation. The aqueous solution being now rendered alkaline by ammonia, the quinine (and most other alkaloids if present) would be precipitated. By a second agitation with ether the alkaloid would be dissolved, leaving in the watery liquid only the sugar, gum, and salt present as impurities, together with the ammonium sulphate produced in the process, and, as this salt is volatilisable, the residue of the evaporated solution when heated should give no black char and no white ash. With a little care and the use of pure barium carbonate, the operation may be satisfactorily modified at the point where the acid is to be neutralised; add barium carbonate instead of ammonia, bring it to the boiling point for a minute, let it cool, and wash out the alkaloid with ether as before described; in this way the water will contain only the barium sulphate and carbonate, both insoluble, and the impurities originally present in the sample. After the separation of the barium salts by filtration, and the evaporation of the clear watery liquor, the residue may be weighed and its nature examined.

In operating on other alkaloids or alkaloidal salts judgment would have to be used as to the acid to be adopted for extraction or solution, and as to the solvent for the free alkaloid.



Returning, however, for the present to the quinine sulphate, to indicate a further small modification of this examination, the  $\zeta$ ss of quinine sulphate shaken with  $\zeta$ ss of pure water, and the latter filtered off, the filtrate would contain any freely soluble impurities the sample might contain, together with the small percentage of quinine sulphate which without additional acid is soluble in cold water; and, as this is only about 1 to  $1\frac{1}{2}$  parts in 1,000, the operation with barium carbonate and ether is more effective with a brief treatment. For this mode of testing alkaloids we naturally prefer to use sulphuric acid as the aqueous solvent, on account of its ready and complete separation when treated with precipitated barium carbonate, but if circumstances required the use of hydrochloric acid this might be subsequently removed by the use of oxide of silver in place of the barium carbonate; or, if oxalic acid were more advantageous with some other alkaloid, there would be a choice of insoluble basic bodies with which it forms insoluble salts.

Treatment by this method can only be held to indicate the absence of a large number of bodies other than alkaloids, and is not adapted to prove the absence of other alkaloids as contaminations of the one under examination. Its chief aim is to detect the presence of extractive matters of the plant which yielded the alkaloid, or to detect chemicals which might be introduced in the process of manufacture.

### ACONITINE

One grain should dissolve entirely in a drachm of water heated to boiling in a test-tube; on cooling, part of the aconitine should be deposited.

This should redissolve on agitation with  $\zeta$ ij additional water without heat. The aqueous solution shaken in a separator with f $\zeta$ ij ether, and the ether after subsidence decanted, this agitation with ether being repeated a second and a third time with fresh portions of ether, should leave the water free from contamination with anything but ether, there being no fixed residue on evaporation of the water.



The three portions of ethereal solution obtained by decantation being now returned to the separator and agitated three successive times with portions of water acidulated with sulphuric acid, each portion of water being carefully run off from the supernatant ether, the ether should now be free from everything but water, and on evaporation should leave no fixed residue ; otherwise fatty, resinous, or caoutchouc-like bodies have been imperfectly removed in the process of manufacture. The uncrystallised aconitine of commerce does not respond to these tests perfectly, and is understood to be an imperfectly prepared alkaloid.

### ADEPS PRÆPARATUS

Lard is frequently faulty from injury done to the fat itself in the process of manufacture, and from the presence of impurities. Lard of commerce was formerly much subject to adulteration with flour and water, but these are now rarely met with.

Cotton-seed oil has recently been the most considerable impurity, and the following test <sup>1</sup> has been recommended by Mr. Conroy (B.P. Conf., 1888).

Make a test solution of—

Silver nitrate, 5 parts.

Nitric acid (sp. gr. 1.42), 1 part.

Rectified spirit, 100 parts.

Melt 100 grains of the lard in a dry test-tube, add 20 minims of the silver solution, and immerse the tube in boiling water for five minutes, taking care that no water enter it. Pure lard remains perfectly white, but if adulterated with cotton-seed oil it takes a brown tint—very pale with 1 per cent., very decided colour with 5 per cent., and very dark with 20 or 30 per cent.

This reducing action on the silver nitrate is probably due to a constituent of the cotton oil which is natural to it, but which may be more or less removed by purification, and the indica-

<sup>1</sup> Known as Bechi's test.

tions of the test are to be regarded not as absolute but as presumptive proof of the purity or the adulteration of the lard.

The official requirement of complete solubility in ether excludes membrane, flour, salt, etc., impurities which, if present, are usually indicated by the melted lard not being clear.

Ether for this purpose should be nearly anhydrous, or the turbidity from separated moisture may lead to the impression that the lard is not totally soluble. Boiling the lard with water and subsequently testing the solution with nitrate of silver is intended to prove its freedom from salt, and the absence of blue reaction with iodine indicates its freedom from flour or other amylaceous matter.

The melting point of lard is not so constant nor so sharply defined as to be of much use in estimating its purity; and its sp. gr. is not so readily taken that the operation would be willingly performed at the dispensing-counter. Food analysts take the sp. gr. of lard at water-bath temperature—from  $210^{\circ}$  F. to  $212^{\circ}$  F.—and state that when pure its density is  $\cdot 860$  to  $\cdot 861$ —the method adopted being to suspend in the hot lard a glass drop which displaces 100 grains of water; the difference between its weight in air and in hot lard is found to be  $86\cdot 0$  to  $86\cdot 1$  grains, giving its gravity without calculation *at this temperature*, and so long as this temperature is uniformly adhered to as a standard this suffices for the purpose intended; but, as the expansion which takes place between solid lard at  $60^{\circ}$  F. and fluid lard at  $210^{\circ}$  F. is considerable, if the gravity of lard in the solid state be required some other means must be adopted. The method used for wax is scarcely applicable, as the lard is not sufficiently firm to bear the handling necessary for weighing, removing air-bubbles, etc. The most convenient process I have tried is to drop melted lard into spirit having a density rather less than the lard should have, say  $\cdot 920$ , and diluting this with weaker spirit, say of about  $\cdot 960$ , till the globules of lard when cold sink or float almost indifferently, and then take the gravity of the spirit with the gravity bottle. In this way I have found the sp. gr. of lard to be  $\cdot 9348$  at  $60^{\circ}$  F. As the spirit is not entirely without action on the lard, this

result must be regarded as an approximation to the truth rather than absolutely correct.

The chief difficulty in this operation is to get rid of air-bubbles, which very readily attach themselves to the surface of the lard, and are very difficult to remove. The plan I have adopted most successfully is to put into a test-tube or glass measure some spirit of greater density than the lard, say about '950 or '960, to pour upon this, without agitation, spirit of '920 or thereabouts, to lift with a dropping tube a small quantity of melted lard and eject it under the surface of the lighter spirit : it will thus form smooth round globules of lard, free from bubbles, which float under the lighter spirit, but on the surface of the heavier. When these are cold the spirit may be mixed, with gentle stirring, so as to avoid carrying air-bubbles down, and then more spirit added of either the higher or lower gravity, as may be required to adjust the density of the liquor to that of the lard. If this final adjustment be effected by the addition of a spirit differing much in strength from that requiring adjustment, trouble will be occasioned by the liberation of air, which always takes place when strong spirit and water are mixed, and this will adhere in a very persistent manner to the surface of the lard, but if the spirit differ but little in strength this liberation of air does not take place.

### ÆTHER. ÆTHER PURUS

The impurities to be anticipated in ether are water, alcohol, aldehyde, acetic acid, sulphurous acid, and empyreumatic oils—the three latter only in crude samples.

The B.P. requires that ordinary ether should contain not less than 92 per cent. of pure ether, while the U.S.P. admits as much as 26 per cent. of alcohol and water. The correct gravity is the one point that should be specially attended to, as it sufficiently indicates that there is not more than the permitted amount of alcohol or water. Freedom from fixed residue on evaporation of f3ij on a watch-glass is the next most important point, though



failure in this requirement is much less frequent than the presence of volatile impurities.

'Ether,' B.P., should have a sp. gr. of  $\cdot 735$ , and 'pure ether' should be  $\cdot 720$ . The latter should make a clear solution with oil of turpentine in all proportions ; the former makes a milky mixture, which, after standing some time, becomes clear from the deposition of the separated water and spirit upon the sides of the vessel. The milkiness is very conspicuous on first mixing, even when the separated water is so small in quantity as to escape observation after standing.

Ether which is absolutely free from alcohol and water does not attack metallic sodium, but this is a degree of purity not to be looked for in a commercial product.

### ÆTHER ACETICUS

Acetic ether may contain as impurities alcohol, water, and common ether. The official tests—that is, gravity ( $\cdot 900$ ) and boiling point ( $166^{\circ}$  F.)—are the points generally relied upon for indicating its quality, and a sample answering to these requirements will generally be satisfactory; but if there were reason to doubt the specimen these indications would scarcely be conclusive. Absence of fixed residue might be added, and its intersolubility with benzine or some other kindred body will probably, when fully investigated, prove a convenient and useful characteristic. The B.P. says it is soluble in all proportions in rectified spirit and ether ; the U.S.P. adds chloroform as a body with which it is intersoluble in all proportions. I have found the intersolubility with oil of turpentine insufficiently sharp to be useful, but with benzol the results were more promising. With a commercial sample from a good source 10 vols. of the acetic ether dissolved benzol, added by degrees till the benzol exceeded 10 vols. ; on making up the mixture to 15 vols. of each the clearness was restored, and on adding 1 vol. of water it again became milky, and speedily 4 vols. (of moist acetic ether) settled out. This can only be regarded as a tentative result.

## ALCOHOL ETHYLICUM

In practice the testing of absolute alcohol is usually confined to an examination of its gravity, its only common impurity being water. The repeated rectification required to remove the water pretty well insures its freedom from fusel oil; methylic alcohol and aldehyde might still, however, be present.

Pure alcohol is so difficult to produce that the B.P. allows from 1 to 2 per cent. of water, giving  $\cdot 800$  as the greatest permissible density. The presence of a little ether in a weaker spirit might bring the density to this point, hence the necessity of testing for water by agitating the alcohol with sulphate of copper which has been reduced to a greyish-white powder by heat; this assumes its common blue colour if the alcohol contains water. To be a delicate test, it should be used only in small proportions, say 1 grain of the dry sulphate to 3i of alcohol.

A more generally convenient test for the pharmacist to use is to mix f 3i of the alcohol with an equal bulk of oil of turpentine, which gives a clear solution with good alcohol, but a milky one with a weaker spirit. It has the advantage of giving its indication immediately and unequivocally, while the action of the dried sulphate of copper is so much influenced by quantity and time as to be inconvenient in its use. With rectified spirit, mixed in the proportion above indicated, the sulphate gradually becomes blue, but a sample of commercial 'absolute' alcohol, which gave no indication of change of colour in a few hours, gave a partial change in 24 hours, and a further development in several days.

Aldehyde and methylic alcohol are both strongly coloured by the action of caustic potash; an alcohol which does not become brown within a few minutes when mixed with an equal volume of solution of potash may be considered free from both these impurities.

The table on next page shows the specific gravity of various mixtures of alcohol and water. In using such tables it is, of course, necessary to make sure of the absence of other bodies, such as sugar and extractive matters, in wine; or ether, amylic or methylic alcohol, in commercial spirits, etc.

SPIRIT TABLE,

*Showing the specific gravity, percentage of alcohol, and excise definition.*

Sp. Gr.	Per cent. Alcohol	
·9981	1	98 U.P.
·994	$3\frac{1}{2}$	95 "
·9914	5	91 "
·988	7	85 "
·9841	10	80 "
·9778	15	72 "
·975	17	66 "
·9716	20	59 "
·968	23	54 "
·9652	25	50 "
·962	27	45 "
·9578	30	38 "
·952	$33\frac{1}{2}$	31 "
·9490	35	27 "
·944	$37\frac{1}{2}$	23 "
·9396	40	18 "
·933	43	12 "
·9292	45	$8\frac{1}{2}$ "
·925	47	5 "
·920	49	Proof
·9184	50	$\frac{1}{2}$ O.P.
·914	$51\frac{1}{2}$	5 "
·9069	55	$10\frac{1}{2}$ "
·900	58	15 "
·8956	60	$19\frac{1}{4}$ "
·890	62	24 "
·8840	65	28 "
·876	68	34 "
·8721	70	38 "
·868	$71\frac{1}{4}$	40 "
·8603	75	45 "
·852	$78\frac{1}{2}$	48 "
·8483	80	50 "
·842	$82\frac{1}{2}$	54 "
·838	84	56 { B.P. Rect. Spirit
·8357	85	57 "
·825	89	63 "
·8228	90	
·8172	92	
·8089	95	
·8031	97	
·7938	100	



## ALUMEN

Common alum of commerce is usually very nearly pure ; it may be either a potash or ammonia salt, and these are used indifferently for most purposes. Iron is the common impurity, either as ferrous or ferric sulphate, and either may be detected by blackening with tincture of galls or glycerine of tannin—the ferric salt most strongly so—and either of them more strongly on the addition of acetate of ammonium. The ferro- and ferri-cyanide of potassium are also used as tests for iron, but have in this instance no advantage over the tannin. Caustic potash causes precipitation of alumina, totally soluble in an excess. Some earths and metallic oxides if present would be left as insoluble in this reagent ; lead and zinc would be dissolved by the excess of potash—they are improbable impurities, but the U.S.P. directs their absence to be proved by there being no precipitate produced on adding a drop of sulphhydrate of ammonium to the solution containing excess of potash. Zinc would produce a white precipitate, lead a black one.

## ALUMEN EXSICCATUM

Dried alum should answer the same tests as the crystalline, except as regards its physical condition and slower solubility. If the heating has been carried too far, a portion of sulphuric acid may be driven off, and some of the alumina being set free is insoluble in water. The slow solubility of the correctly prepared article must not be mistaken for the insolubility produced by overheating. That which is slowly soluble in cold water is quickly soluble in hot. A teaspoonful of dried alum with a teaspoonful of water heated in a test-tube forms a clear solution before it boils.

## AMMONII BENZOAS

The probable impurities are a little aromatic oil, free benzoic acid, resulting from loss of ammonia in evaporation and drying, and, less probably, fixed bases and cinnamic acid.

If there be no excess of benzoic acid 1 drachm should dissolve readily in 6 drachms of cold water, and no appreciable fixed residue should be left on heating a little of the salt on clean porcelain or platinum.

The U.S.P. directs examination for cinnamic acid by treating it with permanganate of potassium, which with this latter acid develops an odour of bitter almonds.

### AMMONII BROMIDUM

The probable impurities in ammonium bromide are bromate, fixed bases, traces of chloride or iodide, and, less probably, sulphate or nitrate. Complete volatilisation below red heat excludes all the probable basic impurities. The presence of bromate is indicated if the action of dilute sulphuric acid immediately develops a yellow colour and pungent odour. The presence of iodate or iodide along with bromate might develop a yellow colour, or brown if the quantity of the iodine compound were more than a trace. If the iodide were present without bromate or iodate, the iodine colour would not be developed except on the addition of an oxidising agent along with the acid; a minute particle of potassium chlorate answers well, and if iodine be present it will be liberated before the bromine, but in the absence of iodine bromine will be set free. It is usual to discriminate by testing with starch, with which a very minute trace of iodine gives the characteristic blue colour. If starch paste be not at hand, a drop of benzine shaken with the solution answers well, and takes a pale pink colour if the iodine be in very small quantity, and a brown if the proportion be larger.

The volumetric test as described in B.P. would give the final reaction too soon if any considerable quantity of iodide were present, and would not give it soon enough if chloride were the impurity; but a combination of these two impurities might give the correct volumetric result.

The absence of chloride is most satisfactorily indicated by precipitating with nitrate of silver in the presence of a little free ammonia, which would retain any chloride of silver in

solution, the bromide and iodide being removed by filtration ; a free addition of nitric acid to the filtrate would throw down any chloride of silver which might be present, together with a *trace* of bromide, the latter being slightly soluble in ammonia. See further under Bromide of Potassium.

### AMMONII CARBONAS

The impurities to be anticipated in this salt are empyreumatic bodies, which become more distinctly perceptible when the salt is treated with an excess of odourless acid, such as sulphuric or tartaric. If the salt be in an effloresced condition it will probably contain bicarbonate of ammonium, and would be deficient in its power of neutralising acids. Fifty-two grains should neutralise 63 of oxalic acid : this is an unnecessary test if the carbonate be in clean compact lumps. Chloride and sulphate are possible impurities, not commonly present ; they may be detected by silver nitrate and barium chloride in solution of the carbonate after decomposition with an excess of nitric acid.

The U.S.P. also adds a test for the heavy metals (ammonium sulphhydrate), but they are not likely impurities. In a salt evidently the product of sublimation fixed impurities are not to be feared, and are not worth testing for, except in a sample in such an effloresced and powdery condition as would naturally cause its rejection.

### AMMONII CHLORIDUM

This is usually a very pure salt ; the only contaminations to be anticipated are a little water in the granular salt, or a little iron in the massive variety as produced by sublimation. As volatility at an elevated temperature is a common property of chlorides, chloride of iron, and indeed other chlorides, might be present, though in practice iron is the one found, and that generally only as a yellowish vein which is sometimes seen running through the masses of the commercial salt. The absence of the heavy metals generally may be proved by the absence of a darkening of a solution of the salt on the addition of ammonium



sulphhydrate, and alkaline or earthy bases would be indicated by the presence of a fixed residue after the chloride had been decomposed by an excess of sulphuric acid.

### AMMONII NITRAS

As found in pharmacy this is usually a pure salt, except that, being deliquescent, it is generally damp.

Neutrality to litmus, the absence of chlorides and sulphates, as indicated by silver nitrate and barium chloride producing no precipitates in a dilute solution acidulated with nitric acid, and the complete dissipation by a gentle heat (below that at which tin melts) are satisfactory indications of the absence of any ordinary impurity. Ammonium chloride is the impurity most to be feared, and is provided for as above.

### AMMONII PHOSPHAS

This salt can scarcely be said to be subject to impurity, the most probable defect being a deficiency of ammonia from loss in evaporation. The salt should have a faint alkaline reaction. The official test with magnesium sulphate is scarcely necessary, and is troublesome from the considerable amount of washing the precipitate requires to ensure its purity, and from its requiring to be washed with a solution of ammonia to prevent loss which would result from the precipitate being somewhat soluble in pure water.

### AMYL NITRIS

The information in B.P. regarding this compound is not satisfactory. The instruction is to produce it by the action of nitric or nitrous acid upon amylic alcohol, which volatilises between  $262^{\circ}$  and  $270^{\circ}$  F., and the product, which is named 'nitrite of amyl,' is to consist chiefly of nitrite of amyl. Presumably 70 per cent. should be nitrite of amyl, as this quantity is required to distil between  $194^{\circ}$  and  $212^{\circ}$  F. Under these circumstances it would not be desirable to attempt any rigid system of testing. It is stated by B.P. to be soluble in all proportions in rectified spirit.

The U.S.P. adds solubility in all proportions in ether, chloroform, benzol, and benzin (petroleum spirit), and both pharmacopœias agree in stating that it is insoluble in water.

### AMYLUM

Starch, according to B.P., may be the product of wheat, rice, or maize, and for all practical purposes it matters very little which of these or of several other varieties be used. Arrowroot, potato starch, or *tous-les-mois* might be mixed or substituted without any important result ; there is no chemical characteristic by which to distinguish them ; the microscope alone can be relied upon.

Whatever variety of starch is used should be purely white. For laundry purposes it is commercially supplied tinted blue ; this should not be used in pharmacy.

The only chemical examination officially directed is testing water with which starch has been gently triturated without the application of heat ; the absence of coloration of the filtered cold infusion on the addition of a drop of tincture of iodine indicates that no granules have been rendered soluble by physical or chemical injury. Rubbing in a mortar with considerable pressure is sufficient to break and render soluble a number of the granules, in which case the filtrate gives an intense blue with the iodine. One drachm of starch stirred with an ounce of cold distilled water and filtered should on evaporation of the filtrate leave a scarcely appreciable residue, and a small portion of the starch ignited on platinum foil should burn away with an almost invisible ash. It is rarely necessary to put commercial starches through any of these tests.

In testing a commercial sample of starch powder I have found, when 3i was shaken with fʒi cold water and then filtered, the filtrate on evaporation left 0·2 residue, and a drachm of the same sample heated to redness, and the charcoal treated with an ounce of water, filtered and evaporated, left a residue of 0·05.

## ANTIMONII OXIDUM

The probable impurities in this preparation are iron, arsenium, and oxychloride of antimony. Copper, lead, and tin are less probable impurities.

As the chloride and some other salts of antimony are decomposed when treated with water, advantage is taken of the tartrate being free from this peculiarity to facilitate the testing.

The oxide should dissolve completely in solution of tartaric acid, and the addition of ammonium sulphhydrate should first produce an orange precipitate, and a further addition should redissolve the same ; a black precipitate would most probably indicate iron, less probably either copper or lead.

Arsenium is not so simply detected, as its reactions very closely resemble those of antimony. A grain of the oxide dissolved in f3i dilute hydrochloric acid, and heated with a small piece of clean copper wire or foil, will impart to the latter a grey or black film, which will contain antimony and arsenium if the latter be present. To determine this point, the copper being gently washed, and dried and heated in a clean, dry test-tube, the appearance of a ring of white sublimate in the tube just above the hot part would suggest arsenium in the form of arsenious acid, and if under the microscope this is seen to consist of octahedral crystals arsenium may be considered to have been conclusively proved.

The presence of oxychloride of antimony in the oxide would be indicated by the solution in tartaric acid giving a precipitate with silver nitrate insoluble in an excess of nitric acid.

## ANTIMONIUM NIGRUM PURIFICATUM

Native sulphide of antimony as it comes from the mine is usually mixed with quartz and sulphides of several other metals—iron, arsenium, lead, etc. As it comes into pharmacy iron is the metal to be expected, the presence of which does not condemn a sample supplied as ‘purificatum.’ The presence of arsenium would of course condemn it, and for the detection of



this there is no better process than that described in B.P. Dissolve a grain or two by boiling in strong hydrochloric acid in a test-tube ; when the odour of  $H_2S$  is driven off, add a little water, not sufficient to precipitate the oxychloride of antimony ; throw in a small piece of bright copper wire or foil and keep it warm for five or ten minutes ; wash the copper in water, taking care not to remove the dark film which will have deposited upon it ; dry it with a gentle heat, and then heat it in a clean, dry test-tube. If any arsenium be present it will become oxidised to arsenious acid at a temperature below redness, and will collect as a white sublimate in the cooler part of the tube, and by the aid of a microscope will be seen to consist of octahedral crystals.

This use of copper and hydrochloric acid is known as Reinsch's test for arsenium ; it is simple, delicate, and has a wide applicability.

If it be desired to examine the sulphide of antimony for iron, a grain or two dissolved in the same way and treated with an excess of ammonium sulphhydrate will probably give a black precipitate, which may contain iron, lead, and copper, the sulphides of antimony and arsenium being retained in solution by the excess of the reagent ; the black precipitate, after washing with water containing a trace of ammonium sulphhydrate, may be treated with a slight excess of dilute hydrochloric acid, which will immediately dissolve any iron it may contain. The solution decanted may be tested for iron with ferricyanide of potassium. The black precipitate, if any remain undissolved, may be dissolved by boiling in strong hydrochloric acid, after which the addition of ammonium carbonate in excess will give a white precipitate if lead be present, and a blue liquor if it contain copper.

### ANTIMONIUM SULPHURATUM

The proportion of sulphur, antimony, and oxygen may vary in this preparation ; to meet this chance the B.P. defines that when 60 grains are thoroughly oxidised by nitric acid, dried and ignited, it should leave 40 grains of oxide of

antimony. The oxide fuses at a red heat and is liable to loss by volatilisation ; the operation is not worth the performance, as the sulphurated antimony is not subject to sophistication, and is of no great medicinal importance. There is no probability of its containing any of the heavy metals but arsenium. That may be sought for by the same treatment as described under Black Antimony, or more simply by digesting the sulphurated antimony in ammonia, filtering off the ammonia, which would dissolve any sulphide of arsenium, but not sulphide of antimony. The ammoniacal liquor if acidulated with hydrochloric acid will deposit sulphide of arsenium if such were present in the antimony compound ; if arsenium had been present in any other combination, which is not probable, the acidulated liquor would yield a yellow precipitate with  $H_2S$ , or might be tested by Reinsch's method with metallic copper.

### ANTIMONIUM TARTARATUM

As found in pharmacy this is usually a very pure salt. The official tests with  $HCl$ , precipitating oxychloride of antimony if the proportion of potassium tartrate be not in excess, and the requirement that 29 grains of the tartar emetic shall yield 15.1 grains of sulphide of antimony when precipitated with sulphuretted hydrogen, both tend to show that the requisite proportion of antimony is present, but are tests scarcely demanded unless there is reason to suppose the sample in hand is a faulty one. A commercial article made for mordanting might by chance find its way into pharmacy ; it usually consists of oxalate of antimony and potassium, but this is not a probable contingency with an article so cheap as tartar emetic. The more probable shortcoming of the pharmacist's stock is deficiency of strength from a little excess of cream of tartar, or excess of strength from loss of water of crystallisation. These variations are usually of but small amount, and if it be desired to have a check upon them a ready means is to ascertain the amount of iodine absorbed in the oxidation of the antimony to antimonie acid. Sixteen grains of tartar emetic

with 12 grains of iodine<sup>1</sup> and 20 of bicarbonate of sodium, rubbed in a mortar with two drachms of water, should leave a colourless solution when the effervescence has gone off, and half a grain additional iodine should render it brown. By this means I have found in a parcel of tartar emetic which had become partially effloresced the opaque crystals indicated nearly 2 per cent. greater strength than those which were transparent.

### AQUA DESTILLATA

Distilled water of absolute purity is not practically known ; its solvent powers are so wide that if it were obtained it could not be preserved and used without contamination. In pharmacy, however, we have to do with what is practicable, and to consider what are the probable impurities which would interfere with its use. When freshly distilled it frequently has an unpleasant odour from the presence of organic matter in the water from which it has been produced. This is not usually sufficient to interfere with any of its uses in pharmacy, and the odour disappears if it is exposed to the air ; but the aëration which makes it more potable introduces other impurities—carbonic acid, oxygen, ammonia, etc.

The B.P. directs that a fluid ounce evaporated in a clean glass capsule leaves a scarcely visible residue. The degree of visibility which it is intended to allow is left to the judgment of the user. The quantity of a residue too small to be weighed may be approximately estimated as pointed out in the chapter on Manipulation ; see 'Fixed Impurities.' A fluid ounce of water leaving less than  $\frac{1}{1000}$  of a grain of residue would be considered good in this particular for pharmaceutical use, and, though a higher purity might be attainable, the total absence of a visible residue could never be accomplished, as water is not entirely without action upon glass.

Carbonic acid is a universal impurity in distilled water, but

<sup>1</sup> Theoretically, 16·2 of tartar emetic should require 12·5 of iodine, so in the first reaction the tartar emetic will be 0·2 in excess, and in the end the iodine will be 0·3 in excess.



one which scarcely troubles us except in the preparation of lead lotions ; the B.P. test with lime-water is not of much use, as carbonate of calcium is appreciably soluble in lime-water or in water holding carbonic acid in solution ; so, to insure the test acting satisfactorily, it would be necessary to add just so much lime-water as would suffice to combine with the carbonic acid which the distilled water contained, and this with the unknown quantity is not practicable. Subacetate of lead is a very delicate test for free carbonic acid, and good distilled water, even after recent boiling and cooling in vacuo, rarely fails to give a precipitate with this reagent.

Ammonia is another common impurity, and is to be sought for, by the official test, with a solution of potassio-mercuric iodide, which gives a yellow or brown colour, according to the quantity of ammonia present. And, as the test is very delicate, and the distilled water very apt to contain a trace, either as a product of the original water or possibly as a contamination from the air of the pharmacy, the B.P. allows that a bulk of three or four ounces may give a visible yellow tint with this reagent. This is known as Nessler's test, and Nessler's solution may be made by the following formula :—

R. Hydrarg. bichlor.	.	.	.	.	.	.	.	ʒi
Pot. iodid. .	.	.	.	.	.	.	.	ʒii ʒi
Aq.								ad ʒiss
Solve; et adde:—								
Potassæ hydratis	.	.	.	.	.	.	.	ʒvi
Aq.								ad ʒivss

The published formulæ for Nessler's solution vary in several particulars ; the essential points are that it should be a strongly alkaline solution, and that the solvent power of the iodide of potassium should be fully saturated with mercuric iodide. Compare B.P., Attfield, etc.

In estimating traces of ammonia by Nessler's reagent it is customary to judge the amount by a comparison of the colour produced with that produced in another solution in which the quantity of ammonia is known.

Distilled water is also required to stand testing with H<sub>2</sub>S,

showing absence of lead or copper which it might acquire from the still. Sulphydrate of ammonium answers equally well. Nitrate of silver, barium chloride, and ammonium oxalate should give no evidence of chlorides, sulphates, and lime, and the official test with starch and iodide of potassium should not indicate nitrite, or other oxidising agent, which might be introduced by adding permanganate to the water in the still for the purpose of destroying organic matter before distillation. The U.S.P. adds a test with permanganate to indicate the absence of organic matter or other reducing agents. 100 parts of distilled water heated with 10 of dilute sulphuric acid, and then coloured pink with a dilute solution of permanganate, should not lose its colour by boiling 5 minutes, nor by standing 10 hours not exposed to the air.

### AQUA LAURO CERASI

The only test to which it is necessary to subject cherry-laurel water is to ascertain that it contains just about 0.1 per cent. of hydrocyanic acid ; this is readily effected by adding f 5i liq. potassæ to ʒiiss of the water, and then 2 grains of silver nitrate. This should form a clear solution if the acid be not deficient, and the addition of  $\frac{1}{4}$  grain more nitrate should cause a permanent precipitate if the acid be not in excess.

### ARGENTI ET POTASSII NITRAS

The test most important in this case is to ascertain that the proportion of silver nitrate is correct. The official process is to precipitate the silver chloride from 30 grains of the mild caustic, and ascertain that its weight after washing and drying is 8.44 grains.

A readier method is to dissolve 50 grains in an ounce of water; add ʒss nitric acid and 5 grains of ammonium chloride; after the precipitate has subsided add half a grain more of the chloride, which should cause a further precipitate; <sup>1</sup> the precipi-

<sup>1</sup> The theoretical quantity of ammonium chloride is 5.22. It is a reasonable latitude to allow it to be between 5.0 and 5.5.

tation this time being complete, a further addition throws down no more silver chloride unless the caustic is stronger than the official standard.

### ARGENTI NITRAS

Crystallised silver nitrate is usually a very pure salt, but the fused caustic in sticks or points may contain potassium nitrate or silver chloride, which may be added in small quantity to give them a tougher character.

The official test by precipitation with excess of HCl, collecting, washing, drying, and weighing the precipitate, affords the means of ascertaining the purity of the salt or the amount of its dilution.

Another method of examination is to dissolve a few grains of the silver nitrate in a few drops of distilled water ; add HCl in excess, and boil ; after subsidence the clear liquor should evaporate on a slip of glass with a scarcely appreciable residue. Examining a tough caustic point in this way, I found 11 grains yielded a residue of 0.3, but, as tough caustic is not supposed to be a pure chemical, a result of this kind is not to be objected to. It is possible, though not probable, that the silver salt may contain lead or mercury, but if it be treated as above the chlorides of these metals as well as others will be found in the clear liquor from which the chloride of silver has subsided, the chloride of lead being pretty freely soluble in hot water.

Theoretically, 17 grains of silver nitrate should exactly precipitate 5.35 grains of ammonium chloride, but, as the article to be tested has an equivalent three times as great as the reagent, a small defect would be easily overlooked if the examination were conducted as suggested under Argent. et Pot. Nit.

### ARGENTI OXIDUM

The probable impurities are silver carbonate, calcium carbonate, traces of earthy or alkaline nitrates, oxides of other metals, and water.

Eleven and a half grains boiled with f 5i dilute nitro-hydro-



chloric acid should become white ; the clear liquor on evaporation should leave no appreciable residue, and the chloride of silver formed should weigh  $14\frac{1}{4}$  grains. If the acid liquor has been found to leave no residue, the chloride may be dried in the vessel in which it has been produced, without washing or collecting on a filter paper. All probable impurities would be dissolved by the acid, and if one drop of the acid liquor leaves a notable residue the amount of impurity may be estimated by washing the chloride and evaporating the acid liquor and washings ; the residue would represent the impurities.

The quantitative test of the B.P.—29 grains of the oxide being reduced to 27 grains of metallic silver by ignition—is scarcely advantageous, from the small difference between the weight of the silver and its oxide.

### ARGENTUM PURIFICATUM

The official tests require silver to be completely soluble in nitric acid (tin would remain undissolved as peroxide), and the solution should not be rendered turbid or coloured on adding excess of ammonia (lead and iron would be precipitated, nickel and copper would be indicated by their colour). Ten grains of the silver dissolved in nitric acid and water, and precipitated by excess of HCl, should on washing and drying weigh 13.25 grains. This would indicate the absence of zinc and some other metals not detected by the ammoniacal treatment. A simpler method is to operate as for nitrate of silver, when the 10 grains or any other convenient quantity of silver is dissolved in nitric acid, and the solution treated with excess of HCl. If a drop of the acid liquor evaporate on a slip of glass without an appreciable fixed residue, purity is sufficiently established, and the collecting, washing, drying, and weighing of the chloride need not be proceeded with.

### ARSENII IODIDUM

The probable impurities are metallic arsenium or its suboxide if the sample has been produced by the trituration pro-

cess, or a contamination of arsenious acid if the salt has been obtained by the evaporation of solution of arsenious with hydriodic acid.

If the former impurities are present they remain undissolved as a dark grey powder when the iodide of arsenium is heated with water; if arsenious acid be the impurity it remains as a white powder very difficult of solution in water.<sup>1</sup> In either case, so soon as the orange-coloured iodide of arsenium has dissolved, the insoluble matter may be separated by filtration, and after washing with a little cold water the usual tests for arsenium or arsenious acids may be applied. The complete or nearly complete volatilisation of the sample on heating in a test-tube excludes many other impurities, but, while these tests cover the likely faults in the preparation, they are not by any means exhaustive.

#### ATROPINA. ATROPINÆ SULPHAS

No very satisfactory tests for the purity of these preparations have as yet been formulated. The probable impurities are colouring matter and some other principles of the belladonna, not perfectly separated by the process of manufacture. One grain of the free alkaloid should dissolve on boiling in a test-tube with a fluid drachm of distilled water, and on cooling should be almost all deposited again, a drop of the clear mother liquor evaporated on a clean slip of glass leaving a scarcely palpable residue. If more than a trace of residue is left, the bulk of the solution should be decanted and tested with chloride of barium, in case the alkaloid has been contaminated with its sulphate. The alkaloid deposited on the cooling of the hot solution, when drained and dried, should dissolve freely on adding a few minims of chloroform; or, if 4 or 5 minims of chloroform be shaken with the watery liquor and the deposited alkaloid, and allowed to settle, both chloroform and water should be clear solutions.

To examine the sulphate of atropine a reverse proceeding

<sup>1</sup> Compare paragraph on solubility in the chapter on Manipulation.

may be adopted: 1 grain of the salt agitated with f 5i chloroform and filtered out; the chloroform should evaporate, leaving a scarcely visible residue. More than this might indicate free alkaloid or some extraneous body. The grain of sulphate of atropine after the adhering chloroform has evaporated should dissolve promptly and completely in a few drops of water, and if boiled in f 5i water with 2 grains of pure barium carbonate and f 5i chloroform added when cold, the whole thrown upon a filter, which removes the barium sulphate and any excess of carbonate, the chloroform should contain all the alkaloid, the aqueous layer should be almost pure water, and on evaporation leave a scarcely visible residue. These tests, it must be remembered, do not guard against the presence of some other alkaloids.

### BISMUTHUM

The probable impurities in bismuth are arsenium, iron, nickel, copper, lead, silver, and tellurium.

Arsenium and tellurium are the most frequent in the bismuth preparations of pharmacy, and usually in so small quantity that they may easily escape detection.

For the examination of the metal, a portion may be dissolved in equal volumes of nitric acid and water, the solution evaporated till, on cooling, it yields a crop of crystals; the mother liquor (A) may be drained off for testing, as it will contain the bulk of the impurities. If (A) gives a precipitate with HCl, silver or lead is indicated; if lead be present it will give a white precipitate also with sulphuric acid. A portion of (A), with the addition of citric acid, and then an excess of ammonia, should give a clear solution free from blue colour, indicating the absence of copper. This ammoniacal solution, (B), treated with excess of sulphhydrate of ammonium, will throw down a black precipitate (C), the mother liquor (D) from which may be separated by filtration, and will contain any arsenium and tellurium, if such be present—also, though less probably, antimony and tin, all of which will be thrown down as sulphides on acidulating the liquor with HCl. If the precipitate produced



by HCl be white, it will be sulphur only, or containing only traces of the metals. If the colour be yellow, arsenium or tin is indicated; an orange colour would show antimony, and tellurium would give brown. If two or more of the metals be present, the darker sulphides may, of course, so obscure the others as to render their presence or absence uncertain without a special examination. To a manufacturer desirous of removing the impurities it would be important to determine their nature absolutely, but to the pharmacist, whose policy is simply to refuse a sample which contains any of these, the complete investigation is scarcely worth the necessary time and trouble. The precipitate (C) will contain bismuth, together with lead, silver, and copper, if these have been detected in the first part of the examination, and may also contain iron and zinc. To determine these two latter, the precipitate should be treated with cold dilute hydrochloric acid, the acid liquor separated by filtration from the insoluble sulphides, and again rendered alkaline with ammonia: a white precipitate will indicate zinc, a black one iron, or possibly both iron and zinc.

### BORAX

Borax of pharmacy is so generally a pure salt as to call for no critical examination.

Commercially, borax is sometimes sold for washing purposes adulterated with carbonate of soda, an impurity which is not anticipated in the official characters, but which would be indicated by effervescence in carrying out the volumetric examination as the B.P. directs. According to the Pharmacopœia, a drachm of borax, dissolved in 3 ounces of water, should be 'saturated' by  $19\frac{3}{4}$  grains of oxalic acid. The point of neutrality is not sharply indicated by litmus, and the test is scarcely worth performing, except as an indication of the absence of carbonate. The only other point which is at all likely to be indicated by the volumetric examination is deficiency of water. Borax may be crystallised with less than ten molecules of water, or it may lose some of its water of crystallisation by keeping.

## BROMUM

Bromine may be contaminated with iodine or chlorine. The B.P. test for the former is simple and effective ; iodine, if present, is last to combine, or first to be liberated from combination, with the solution of soda, with which the bromine is directed to be shaken, and its existence in a small proportion becomes conspicuous on adding starch or flour paste, as directed in B.P.

Chlorine is not so readily detected, and is a not uncommon impurity in bromine as first liberated from brine in the process of manufacture, though not in large proportion, and not usually appreciable in bromine as it is found in pharmacy. The B.P. gives the sp. gr. and boiling point as general indications of the purity of bromine, but with a liquid of this nature a careful determination of these points is both difficult and unpleasant. Probably most pharmacists would find the least troublesome way of detecting chlorine in bromine would be the conversion of the sample into an iron salt as follows :—Add an excess of iron (filings or wire) to a portion of the bromine in 5 or 6 volumes of water, and when the action has ceased filter the solution, add silver nitrate so long as a precipitate is formed, wash the precipitate to remove the iron and excess of silver solution, throw the precipitate into a weak solution of ammonia, macerate for a few minutes, and filter. Bromide of silver being nearly insoluble in ammonia, and the chloride freely soluble, the ammoniacal solution will now contain, as chloride of silver, any chlorine which might have been present in the bromine under examination, and will show the same as a precipitate of chloride of silver when the ammoniacal solution is acidulated with nitric acid. Compare Potassium Bromide.

## BUTYL-CHLORAL HYDRAS

The impurities to be anticipated are ordinary chloral hydrate and other products of the action of chlorine on alcohol or aldehyde. Neutrality to litmus will mark its freedom from the

acids produced in the primary reaction, and the absence of an odour of chloroform on the addition of solution of potash will show that ordinary chloral is not present. The B.P. gives its melting point as about  $172^{\circ}$  F., and its solidifying point after fusion as about  $160^{\circ}$  F., which may be regarded as a general indication of its being the right substance when taken in conjunction with other characters, but does little towards indicating its purity. Its freedom from fixed residue on heating to water-bath temperature and its behaviour towards solvents are more important points. A drachm being shaken with 2 or 3 drachms of water and thrown upon a filter, a fluid drachm of the filtrate should leave on evaporation (at  $80^{\circ}$  or  $90^{\circ}$  F.) one grain of dry residue (theoretically about 1.08); any considerable excess would indicate some impurities. Treated in the same way with chloroform, a fluid drachm of the filtrate should leave only a small fraction of a grain of residue (about 0.1); any considerable excess would indicate impurities of some other nature. Chloral hydrate, if present, would be found in both these residues. These two solvencies, together with its total solubility in its own weight of glycerine or rectified spirit, may be taken as satisfactory indications of the purity of the sample under examination.

It is also freely soluble in ether.

### CAFFEINA

The B.P. gives a general indication of the absence of other alkaloids by treating a solution of caffeine with an excess of tannic acid in which the caffeine at first precipitated redissolves, while most other alkaloids would remain insoluble. With the same view the U.S.P. requires that solution of caffeine should give no precipitate with the double iodide of mercury and potassium, which is a very general precipitant of alkaloids.

Five grains of caffeine are not entirely dissolved by agitation with f 3vi of cold water, but freely with an ounce. If this aqueous solution is agitated with half an ounce of ether in a separator, the ether when separated and evaporated leaves no residue. After



separation of the ether, if the aqueous solution be shaken with f ʒij chloroform, this separated, and the shaking repeated with another drachm of chloroform, the water should be deprived of all the caffeine, and on evaporation should not leave more than an insignificant residue of fixed impurities. The most probable impurity is citric acid—from a laxity in the use of the name ; it would be indicated by the greater solubility in water, and by the acid being left in the aqueous solution after the shaking out with chloroform.

### CAFFEINÆ CITRAS

The fault to be anticipated in citrate of caffeine is either the presence of other alkaloids or the deficiency of caffeine. Twenty grains being dissolved in f ʒij water with gentle heat, and ammonia added in slight excess—the whole shaken in a separator with f ʒij ether—the ethereal fluid on separation should evaporate with very little residue. The same aqueous mixture treated twice with chloroform in the same manner, and the two chloroform solutions evaporated together, should yield a dry residue of caffeine, weighing 10 grains. The aqueous solution should have the saline taste of citrate of ammonium, but be almost free from bitterness.

The almost entire absence of ash on incineration is a ready means of proving its freedom from fixed salts, and the precipitate produced by tannic acid in an aqueous solution being completely soluble in an excess of the reagent is an indication of the absence of alkaloids generally.

### CALAMINA PRÆPARATA

Calamine is essentially an impure carbonate of zinc ; commercial specimens vary from nearly pure carbonate of zinc, coloured by the presence of iron, down to others which are calamine only in name, and consist of barium sulphate and colouring matter. Silicate of zinc is also occasionally found in place of the carbonate. The almost total solubility in acids required by the B.P. excludes any objectionable contamination

with or substitution of these, which are the only probable impurities. A more complete, though scarcely necessary, examination would be effected by adding to the clear solution, in hydrochloric acid, ammonia in excess, which should at first precipitate and then redissolve the zinc without colour or residue, and the ammoniacal solution thus produced, when treated with excess of sulphhydrate of ammonium, should give a precipitate nearly white, the filtrate from which should leave on evaporation only a residue of ammoniacal salts, entirely volatilisable below red heat.

### CALCII CARBONAS PRÆCIPITATA

The most probable impurities are traces of chloride of sodium, chloride of calcium, or carbonate of sodium, present from imperfect washing ; also, less probably, calcium phosphate or sulphate, magnesia, alumina, and oxide of iron, derived from the limestone from which the chloride of calcium has been produced.

If  $\frac{3i}{\text{of}}$  precipitated chalk be shaken with  $\frac{3i}{\text{of}}$  distilled water, and the water after filtration gives no precipitate with nitrate of silver, there is scarcely need to go further with the testing. The other impurities not soluble in water are detected by dissolving  $\frac{3i}{\text{of}}$  of the chalk in  $\frac{f 3i}{\text{of}}$  of dilute nitric acid ; this should make a clear solution, which, after boiling to expel carbonic acid, yields no precipitate on adding saccharated solution of lime to an alkaline reaction. A precipitate formed under these circumstances would almost certainly indicate one or other of the impurities in the second list above. The B.P. directs the use of nitric acid for dissolving the precipitated chalk, that the solution may be tested at the same time for chlorides.

If chlorides have been sought and found absent by the treatment with distilled water, dilute hydrochloric acid may be substituted for nitric acid with at least equal advantage, and in this case ammonia may be used as the precipitant instead of saccharated solution of lime. The latter is, however, the more certain of indicating magnesia if present.

A trace of calcium sulphate, which is a very probable though not a very important impurity, is not provided against by the B.P. tests ; it would be indicated in the acid solution by precipitation with barium chloride. Any considerable contamination with this substance would be seen by its remaining undissolved by the acids.

### CALCII CHLORIDUM

Chloride of calcium, if made by the B.P. process, will not probably contain iron, alumina, or magnesia, which may be supposed to be the impurities to be detected in its solution by the addition of lime-water causing a precipitate. These would be separated by the addition of chlorinated lime and hydrate of lime, which the B.P. directs to be added to the crude solution of calcium chloride. With these additions, however, there is the chance of calcium chlorate being introduced as a result of heat acting upon any excess of chlorinated lime. This impurity would be indicated by the evolution of chlorine or hypochlorous acid on the addition of a little hydrochloric acid to the chloride of calcium.

### CALX. CALCII HYDRAS

The probable impurities in lime are magnesia, soda, alumina, iron, carbonate, phosphate, sulphate, chloride, and silicate of calcium. These are of no practical importance for the preparation of lime-water or saccharated solution of lime, but when used as a basis for other salts, or as a reagent, the chance of these impurities being present must not be overlooked. The sodium salts and other soluble compounds are directed to be washed out before the lime-water is prepared, and the salts insoluble in water do not interfere with the product.

### CALCII HYPOPHOSPHIS

This salt is not much subject to impurities ; the most important indication of its quality is the official test with potassium permanganate : 5 grains of the hypophosphite boiled in half an



ounce of water with 12 grains of the permanganate, and filtered, gives a nearly colourless solution, showing that the hypophosphite has its legitimate reducing power. If this point is satisfactory, the next question is whether this action may be in any degree the result of impurities which have a like property, such as sulphites or hyposulphites. These would be indicated by yielding a white precipitate with the official test with acetate of lead. The B.P. test with perchloride of mercury is intended to indicate the identity of the salt under examination, from its power of reducing the corrosive sublimate first to calomel and then to metallic mercury.

### CALCII PHOSPHAS

Precipitated calcium phosphate rarely contains any impurity of pharmaceutical importance, though I have found it contain carbonate, an impurity so certain of detection as to insure its appearance being only an accident; the chief consumption of the phosphate being in acid solutions, the effervescence which would be apparent in this use would at once condemn the sample. More probable impurities are ammonium and calcium chloride from imperfect washing. These would be detected by agitating 3i of the phosphate with an ounce of distilled water, filtering, and evaporating, which would leave any ammonium or calcium chlorides. The B.P. indicates the absence of iron by a solution in nitric acid not yielding a precipitate on the addition of an excess of sodium or potassium acetate, the phosphate of calcium being soluble in acetic acid, but not the iron salt.

Calcium phosphate being almost exclusively used for the preparation of syrups intended to supply bone material to the patient, traces of bone materials other than calcium phosphate would not in most cases be of serious consequence.

### CALCII SULPHAS

The native calcium sulphate is usually pure enough for all practical purposes. Traces of iron and sodium, which are

so generally distributed in Nature, may be found, and the former is no doubt the source of a greenish-grey tint of the calcium sulphide as sometimes seen, but the quantities are too small to be of any importance for pharmaceutical use.

### CALX CHLORINATA

This is one of the variable chemicals—variable because it is at best an indefinite compound, or mixture of compounds, and there is no means of purifying it. The product may vary in consequence of too little or too much water used in hydrating the lime, and too much heat, or too little exposure to chlorine ; and to a still greater degree is it likely to vary from long or careless keeping. Loss of *available* chlorine and absorption of water are the two points which must have attention. If in a wet, pasty state, it may at once be condemned as unfit for use in pharmacy. The quality as regards available chlorine may be tested as follows : 5 grains being diffused in an ounce of water, add 15 grains of KI and f 3i hydrochloric acid, or sufficient to produce a clear brown solution ; this should continue to have a brown colour on adding 10 grains of hyposulphite of soda, but should be rendered colourless by 2 grains more. The addition of KI serves the double purpose of preventing loss of Cl, by converting the chlorine as it is liberated into KCl, and liberating the iodine which is retained in solution, and serves the purpose of a colour reagent, showing when sufficient hyposulphite has been added to convert it into HI.

For pharmaceutical use the test may be further modified by taking  $11\frac{1}{2}$  grains of the hyposulphite in an ounce of water, adding 2 or 3 drops of tincture of iodine ; pour into this, drachm by drachm, a solution of 10 grains of chloride of lime in 10 drachms of water ; the number of drachms added by the time the first permanent brown colour is produced indicates the number of grains of the sample, which has a strength equal to 5 grains of the official standard. The hypo solution may conveniently be used in a measure with stirring-rod, and the chloride of lime with the water in a phial ; shake repeatedly,

and pour gradually into the hypo with constant stirring; when this becomes brown ascertain how much of the lime solution has been used by measuring the remainder.

### CALX SULPHURATA

Sulphurated lime is a rather uncertain chemical, consisting of sulphate of calcium partially reduced to sulphide of calcium by the action of the charcoal with which it has been calcined, and it is prone to further change by keeping. The odour of sulphuretted hydrogen is probably indicative of the absorption of carbonic acid and the decomposition of the sulphide. The B.P. test—rubbing 8 grains of the sample with 14 grains of sulphate of copper in an ounce of water and a little hydrochloric acid—having been applied, the copper should be converted into sulphide at the expense of the calcium sulphide, and, if this latter is in due proportion, and the action, which is rather slow, has had time to become complete, the filtrate should be free from copper, and consequently give no red coloration with ferrocyanide of potassium. Trituration and heat both expedite the action.

### CERII OXALAS

The B.P. acknowledges the common presence of oxalate of lanthanum and didymium in this salt, and, so long as it does so, their presence in small quantity should not be sufficient to condemn the sample. The oxide of the latter metal is of a dark colour, and its presence may be judged by the colour of the oxides left on ignition of the oxalate at a dull red heat. The oxides of cerium and lanthanum are of a pale yellow or buff if didymium be not present, and the oxide of didymium is said to become dirty white if ignited at a white heat. Iron is a natural impurity of the ore, but is readily removed in manufacture. Lead is also said to be common. Oxalates of calcium, magnesium, and aluminum may be present. The two former would be indicated in the burnt residue, provided the heat has been limited to a dull red, by its effervescence with hydro-



chloric acid, the oxalates having been converted into carbonates by heat. If the oxalate contains alumina, boiling with caustic potash dissolves it, and the addition of ammonium chloride to the alkaline liquor after filtration throws down the alumina. When the oxalate is treated with caustic potash, oxalate of potassium is formed, and the liquor after filtration and neutralisation with acetic acid will give a precipitate of calcium oxalate on the addition of a solution of calcium chloride. This reaction is directed to be used as an indication that the salt under examination is an oxalate. The alkaline solution may also contain lead and zinc, which would be indicated by sulphhydrate of ammonium giving a black precipitate with the former or a white one with the latter. If the sample be tested by treatment with sulphhydrate of ammonium, and is not darkened, it is unnecessary to look further for lead, copper, or iron.

### CERA FLAVA

Yellow wax is frequently impure either from imperfect preparation or subsequent sophistication; in the former case it may contain honey, pollen grains, etc., and in the latter resin, fat, or paraffin is the most likely. Perfect solubility in hot oil of turpentine, as required by the B.P., would exclude most of the former impurities. But starch is very difficult to see in the fluid wax and turpentine, and the examination with boiling water is desirable; the melted wax being shaken with an equal bulk of distilled water, and then allowed to subside, the water should evaporate with a very small residue. Agitation with boiling solution of caustic soda would dissolve resin and fat, and when the alkaline liquor is separated from the wax, and acidulated with hydrochloric or other acid, these bodies if present would be precipitated, giving it a milky appearance. Adulteration with paraffin is best detected by an examination of the gravity and melting-point. The latter may be conveniently observed by rubbing the bulb of a thermometer with the sample of wax so as to coat it with a thin film, placing the bulb within a dry test-tube to form an air-chamber, which may

be gradually heated by immersion in a beaker of hot water. The necessity for this hot air-chamber is simply that the heating and cooling may be so slow and equable as to enable an accurate observation to be made with facility. The film can be conveniently watched through the glass, and the temperature noted at the moment of its becoming transparent, and noted again at the moment of its becoming dull on removal from the hot water. After the wax has once got up to the melting-point the temperatures at which the bulb becomes bright and dull may be observed several times in the course of a few minutes by immersing and removing the test-tube from the beaker; if two observations in succession are concordant, it may be considered satisfactory. The melting and congealing points as thus indicated should be about  $146^{\circ}$  F. and  $143^{\circ}$  F. respectively. Most adulterants tend to lower these.

The sp. gr. of wax is defined to be from 0.950 to 0.970. Most adulterants tend to lower this point also. A handy observation is to throw a fragment of the sample into .959 solution of ammonia, in which it should either sink or swim almost indifferently.

Another ready method is to weigh 100 grains of wax, stick a 5 grain brass weight into it, and it should sink in water, but with a 3 grain weight it should float. The B.P. probably allows a greater latitude than is necessary. I have found 100 grains of pure wax sink with a 4 grain weight attached, float with a 3 grain weight, and about balance with  $3\frac{1}{2}$  grains brass weight, but float with  $3\frac{1}{2}$  grains aluminium weights. Dieterich, in 'Pharmaceutical Journal' (March 1887), says its gravity should be .963 to .964. By whatever method the sp. gr. is taken, it is important to get rid of bubbles of air, which are apt to adhere rather persistently to its surface. Brushing the sample with spirit or solution of ammonia before immersing it in water generally accomplishes this without difficulty.

A good physical indication of the quality of wax is to warm a little fragment in the hand, press it on the ball of the thumb with the forefinger, drawing the finger with pretty strong pressure from the tip of the thumb towards the joint. Good

wax treated in this way should curl up towards the end of the moving finger, while inferior qualities usually either spread like salve upon the thumb or crumble with the friction.

White wax is more commonly impure than yellow, but ought to stand the same tests. The commercial white wax in round cakes, as usually supplied by wax chandlers, I have found to have a density of .939 and a melting point of  $103^{\circ}$  F., congealing again at  $128^{\circ}$  F. This being a candlemaker's product, the same strict regard for purity is not to be anticipated as would be looked for in a white wax supplied as a pharmaceutical product.

### CARBO ANIMALIS PURIFICATUS

The B.P. test with litmus to prove the power of absorbing colouring matter shows that the animal charcoal is in good physical condition, and has not lost its essential property by careless preparation or keeping. Its chemical purity is very satisfactorily indicated by the combustion test, which gives the lowest standard of purity which a manufacturer should work to. But the process is not a convenient one for the dispensing-counter, as the combustion is slow, except under the influence of a powerful heat or with the aid of chemicals, and has the disadvantage of condemning some samples which are practically good, though they contain more incombustible matter than the B.P. allows. I have found a sample, supplied as of B.P. purity, which yielded 9 per cent. of incombustible matter, the greater part of which was silicious sand, and the yield of soluble matter to hydrochloric acid was so small as not to interfere with its use for decolorising purposes. If 20 grains of the animal charcoal be boiled in half an ounce of dilute hydrochloric acid, the solution filtered, and an excess of carbonate of ammonium added, the precipitate after washing and drying should be only a fraction of a grain.

A simpler examination for the pharmacist is to boil 100 grains of the sample in 1 ounce of dilute hydrochloric acid for five minutes, throw this upon a filter, and wash with half an



ounce of water : the filtrate on evaporation should not leave more than half a grain of non-volatilisable residue. This test does not cover the same ground as the combustion test, as it may leave in the carbon a portion of calcareous matter which a thorough operation would remove, and which would all be obtained in the ash of the combustion test, and, besides that, silica may be present in the carbon in a condition which the acid would not attack, and which consequently would not unfit the sample for most of the purposes to which it is applied. The acid test is calculated to detect all the impurities which would disqualify this sample for general use.

### CETACEUM

The only probable impurity in spermaceti is the fluid portion of the sperm oil imperfectly separated by the process of purification. Its presence may be judged as well by the unpleasant odour and oily stain which it imparts to paper in which it may be folded as by any chemical test.

The free solubility of the sample in hot rectified spirit excludes wax and any considerable mixture of paraffin, but is not an indication of any great value ; nor is its melting-point ( $111^{\circ}$  F. to  $122^{\circ}$  F.) or its sp. gr. (about '94), as these are closely analogous to those of sundry other bodies which might be present.

The spermaceti naturally contains several fatty bodies, and cannot be expected to have the constancy of a definite chemical ; but the expression 'freely soluble in hot rectified spirit,' which is used by the B.P., wants better definition if it is not to mislead the pharmacist. Rectified spirit as defined by the Pharmacopœia, alcohol with 16 per cent. of water, will at its boiling-point dissolve about 1.5 per cent. of spermaceti (an experiment gives 1 grain just dissolved by 90 minims = 67 grains of rectified spirit), and this can scarcely be said to be a free solubility. On the other hand, absolute alcohol at its boiling-point continued to dissolve spermaceti added by degrees till the spermaceti was in larger quantity than the alcohol, and there is no doubt that

this is free solubility, and that absolute alcohol is a highly rectified spirit, but it is not *the* rectified spirit of the Pharmacopœia.

### CHLORAL HYDRAS

This, when in the form of clear, loose crystals, soluble in less than their own weight of water, is generally very pure. The official test with strong sulphuric acid acting on a strong solution of chloral in chloroform, giving no brown colour, is intended as an indication of the general absence of organic impurities. The U.S.P. uses the acid without chloroform and warms the mixture (equal bulks of strong sulphuric acid and chloral hydrate), and requires that the mixture shall not blacken.

As chloral may combine with alcohol, forming an alcoholate of chloral, which is of less value than the hydrate, this is one of the points against which the official tests are directed; the alcoholate has greater solubility in chloroform, and the official statement that the sample under examination shall be 'soluble in four times its weight of chloroform' ought rather to have been 'not soluble in less than four times its weight of chloroform.' The alcoholate has also a higher boiling-point and a smaller yield of chloroform than the hydrate. The B.P. gives the boiling-point as 202° to 206° F., and states that it volatilises on platinum foil at a slightly *higher* temperature. This is evidently a mistake for *lower* temperature, as it volatilises gradually at ordinary atmospheric temperatures, and more rapidly as the heat rises. For example, a grain having been placed on the metal scale of a thermometer laid upon a hot plate, the chloral had entirely evaporated before the instrument indicated 140° F. The important point to note is that there is no fixed residue left by the time its boiling-point has been reached.

The yield of chloroform on distillation with water and lime is not a practical test. Most operators would fail to obtain the theoretical yield, and, if their sample of chloral hydrate otherwise appeared satisfactory, would conclude that the fault was in the operation rather than the sample.

## CHLOROFORMUM

Chloroform is very generally found in pharmacy in a very satisfactory condition of purity. The B.P. allows 1 per cent. of alcohol, which is found to improve its keeping quality and is not in any respect pharmaceutically objectionable. Free chlorine, hydrochloric acid, sulphurous acid, aldehyde, and other products of the decomposition of alcohol are impurities only found in badly made samples. A portion of chloroform shaken with an equal volume of water, and the latter tested with litmus, should indicate the absence of acids. The aqueous liquor should not indicate chlorides with nitrate of silver, nor free chlorine by developing a brown colour with iodide of potassium. The chloroform itself, shaken with an equal volume of strong sulphuric acid, should only give a very pale brown colour—comparable with that produced by shaking one minim of spirit with 100 minims of the pure acid. More than this coloration would indicate the presence of other organic bodies. Chloroform shaken with an equal volume of liquor potassæ and warmed should not turn brown, or aldehyde may be suspected, and if iodine be added to the potash solution after it has been separated from the chloroform, and the heat continued, a small precipitation of iodoform should take place, showing that the original chloroform contained the small addition of alcohol which is requisite to prevent its decomposition by keeping.

## CHRYSAROBINUM

In the original issue of the B.P. the characters and tests of this article were inconsistent with the description, and in the corrections subsequently issued the matter was not made so clear and explicit as it should have been. Chrysarobin is still stated to be 'medullary matter dried, powdered, and purified,' and the characters and tests are to apply to the substance after it has been purified by solvents, and it is ambiguous which is to be used when chrysarobin is ordered. The right course would



have been to limit the term Goa powder to the crude material, and the term chrysarobin to the same after purification by solvents.

### CINCHONÆ CORTEX

The assay of cinchona is not under any circumstances a very easy process, or one likely to be repeatedly used by the dispensing pharmacist. As the official requirement is that the cinchona shall yield the prescribed percentage of alkaloids by the particular process described, it would be inappropriate to indicate other methods, even if such were more convenient.

### CINCHONIDINÆ SULPHAS

There is considerable difficulty in completely separating the cinchona alkaloids from one another, and, while the commercial supplies are usually fairly satisfactory, absolute purity is scarcely to be looked for, and the systems of testing are directed to the prevention of any undesirable laxity in their preparation.

In the salt under consideration the B.P. indicates the general absence of cinchona alkaloids other than cinchonidine and quinine by precipitating these two by the addition of a solution of tartarated soda to a solution of the sample, which throws down these, but leaves the others in solution ; the filtrate should yield only a trace of precipitate on the addition of ammonia. The approximate freedom from quinine is then to be indicated by dissolving a grain of the sulphate of cinchonidine in 3ss of water with 3ss of dilute sulphuric acid, which solution should have a scarcely noticeable fluorescence, otherwise quinine is probably present in more than a trace. The absence of many other organic impurities is shown by the cinchonidine being soluble, without more than a faint yellow colour, in strong sulphuric acid, the application of a gentle heat not appreciably increasing the shade. The absence of ash excludes most of the inorganic impurities with which it might chance to be contaminated. A good general indication of the absence of many impurities is to dissolve 10 grains in half an ounce of hot water ;

add 10 grains of pure carbonate of barium and boil for a few minutes ; allow it to cool, and filter off f3j and evaporate ; the residue should weigh less than  $\frac{1}{10}$  of a grain. Shake the aqueous liquor containing the barium and cinchonidine precipitates with ether, and after subsidence a fluid drachm of clear ethereal solution on evaporation should leave not more than  $\frac{2}{10}$  of a grain of residue. This would not prove the entire absence of other alkaloids, but would exclude a great variety of substances which might be accidentally present.

### CINCHONINÆ SULPHAS

This salt is more soluble in chloroform than the sulphates of most other alkaloids. The almost total solubility in 60 or 70 times its weight of chloroform and the almost total insolubility in ether give indications of a probably good salt. A further confirmation is obtained by applying the test for organic impurities with sulphuric acid as described under Cinchonidine Sulphate, and noting the absence of fluorescence and of ash.

### COCAINÆ HYDROCHLORAS

The characters in B.P. are intended to indicate the identity of the salt ; the tests of purity only include four points : freedom from colour when treated with cold concentrated acids (organic impurities), little or no cloudiness with chloride of barium (sulphates) or oxalate of ammonium (lime), and burning without ash (fixed bases).

It has recently been suggested to detect the amorphous alkaloid in hydrochlorate of cocaine by the behaviour of a dilute solution when rendered alkaline with ammonia (see 'Pharm. Journ.' xvii. p. 686 ; 'Chem. & Drug.' xxx. p. 297). Mr. Mac-lagan says 1 grain dissolved in 2 ounces of water, and two drops of solution of ammonia added, will, if the salt be pure, yield lines of minute crystals where the stirring-rod has rubbed the sides of the glass, a flocculent precipitate will fall, and the liquor after subsidence will be bright. With a good commercial sample I

have obtained his results, except that the precipitate was purely crystalline, scarcely to be called flocculent. He adds that if the amorphous alkaloid be present even in small proportion no striæ will be formed on the glass and no precipitate will form, the liquor remaining milky for a long time and having an oily appearance on the surface. I have not met with a sample which has enabled me to confirm these latter statements.

### CODEINA

Codeine is usually well crystallised and of satisfactory purity.

One grain dissolves slowly, but completely, in fʒij of boiling water and deposits nothing on cooling. This solution has an alkaline reaction, and when shaken in a separator with an equal volume of petroleum ether the latter on separation and evaporation leaves no fixed residue. If the aqueous solution be now shaken with fʒiiss chloroform, the chloroform removes the most of the codeine, and after a second shaking with chloroform the aqueous liquor should be nearly free from bitterness and evaporate with a fixed residue too small to be weighed.

### COLOCYNTHIDIS PULPA

Powdered colocynth of commerce must not be mistaken for the official colocynth pulp; the former may be readily distinguished by the grittiness of the powder between finger and thumb, due to the toughness of the seeds making it impracticable to reduce them to a smooth, soft powder. As the seeds are oily as well as tough, the B.P. confirms their absence by requiring that the powder shall not yield oil when treated with ether. Shake ʒss of the sample with fʒij ether, throw upon a filter paper, and evaporate the filtrate. It should not leave a drop of oil.

### CONII FOLIA ETC.

Hemlock and its preparations should have a slight mousy odour, much increased by the addition of liquor of potash. As



the volatile alkaloid coniine is fixed by combination with acid in the plant, a strong odour should not be perceptible till the addition of the alkali has set the alkaloid free.

### COPAIBA

As there is a considerable variability in this oleo-resin, it is not practicable to lay down definite rules for establishing its purity.

The sp. gr. ranges from .940 to .998, and the resinous residue, after long-heating to drive off the essential oil, is not always friable when cold; but a very soft sticky residue would lead us to suspect adulteration with some fixed oil, and an odour of turpentine during the heating would certainly condemn the sample.

The U.S.P. adds the requirement that the essential oil which it yields should not boil below  $200^{\circ}\text{C}$ . ( $392^{\circ}\text{F}$ ), a test much too troublesome for general application, and not altogether reliable. It also adds a test for Gurgun balsam as follows:—On adding one drop of copaiba to 19 drops of disulphide of carbon, and shaking with a drop of a cold mixture of equal parts of sulphuric and nitric acids, it should not acquire a purplish-red or violet colour.

### CREASOTUM

Creasote of pharmacy is not a definite compound, but a mixture of several products of the destructive distillation of wood, and our rule must of necessity be to secure such a product as the official characters and tests demand. The B.P. tests are chiefly directed against insufficient purification of the wood tar-oils, and fraudulent addition of coal-tar products.

If insufficiently rectified it is brown in colour, but even good qualities acquire a brown colour with age. The absence of a coagulating action upon albumen, or collodion, distinguishes it from carbolic acid, and the absence of a permanent greasy stain when a drop is let fall upon paper and exposed to the air at a temperature of  $212^{\circ}\text{F}$ . shows its freedom from fixed oils

and resin. The B.P. is a little ambiguous on this point, through the omission of the word 'permanent' and the omission of the instruction to expose it to the air as well as heat. Any one desirous of applying the temperature of  $212^{\circ}$  F. might naturally put the paper moistened with creasote into a test-tube immersed in boiling water, the requisite temperature being thus secured. But under these circumstances evaporation goes on very slowly, for though evaporation of creasote takes place at atmospheric temperatures when exposed to the air, it is very slow in a confined space even at  $212^{\circ}$  F., for its boiling-point is about  $400^{\circ}$  F. In this particular, Attfield is also wanting in clearness. He says, regarding the discrimination between creasote and carbolic acid, 'that the former *boils* only at  $370^{\circ}$  F. while the latter readily dries up at  $212^{\circ}$  F.' The fact is, both of them when dropped upon paper and exposed to the air dry up readily  $50^{\circ}$  or  $60^{\circ}$  below this temperature. Miller says creasote may be regarded as pure if its boiling-point be constant at  $398^{\circ}$  F., and if it does not become brown by keeping.

## CROCUS

A great deal has been said and written about the sophistication of saffron. It has been found to be adulterated with oil, sugar, deliquescent and earthy matters. Oil may be noticed by its imparting a greasy stain to paper in which it is folded; earths by their settling out from water with which a sample has been shaken; sugar and saline matters by their appearance on evaporating the clear infusion. A general check is also afforded by the ash amounting to about 6 per cent. But even when these are supplemented by an examination of the structure of the filaments, they are scarcely conclusive proof that the drug has not been sophisticated. An examination of its tinctorial power is of more value than any other indication. One grain of saffron, well exhausted and well diluted, gives a colour which closely resembles, both in character and depth, the colour produced by 14 grains of potassium bichromate dissolved in the same bulk of water.

The colour produced by macerating a grain of saffron in fʒj of rectified spirit looks so much paler than an aqueous infusion made in the same way as to give the impression that water is the best solvent, but if these are both diluted with a pint of water, the alcoholic extraction is found to be most highly coloured ; hence we see the necessity for having regard to the degree of dilution ; and if we boil the saffron with successive drachms of spirit till exhausted, and do the same with the aqueous treatment, diluting the total product in each case with water to a pint, we find each pint equally well coloured, but that the spirit has effected exhaustion with less than half the trouble. A grain of saffron boiled with three successive drachms of spirit is fairly exhausted, and it requires seven or eight boilings with water to obtain equally good results. I have found the alternate action of spirit and water sooner completes the exhaustion than either solvent used singly.

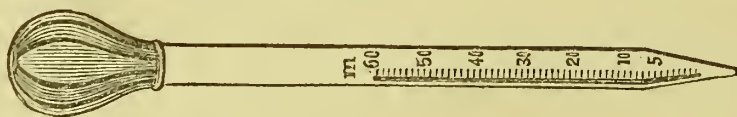
A grain of saffron is a convenient quantity to work upon. This being placed in a phial with fʒij of ether, should yield to the latter a yellow colour, but of no great intensity, otherwise the presence of some of the aniline dyes may be suspected. The ether being decanted, and the phial warmed till the saffron is again dry, fʒij of spirit, rectified or methylated, is poured upon the sample, and heat short of ebullition is then applied for an hour or so ; the tincture is then decanted into a 2 oz. phial. The saffron is next treated with fʒij of water and treated as before, the infusion being added to the previous liquor, and the extraction is continued with alternate portions of spirit and water till the solvents cease to extract colour, and the fibres are nearly white. Three portions each of spirit and water usually effect a pretty complete exhaustion. The total liquors being made up to 2 fluid ounces should have an orange colour, closely resembling a solution of 14 grains of bichromate of potassium in 2 ounces of water, but the true value of the colour cannot be critically judged unless the liquors are very considerably diluted. I have found the most satisfactory results obtained by taking 8 minims of the standard bichromate of potassium liquor and diluting it with an oz. of water, half filling a test-tube



of about half inch diameter with this dilute standard, then adding to an ounce of water 8 minims of the saffron liquor, and having half filled a similar test-tube with this, holding the two tubes side by side against a sheet of white paper, standing with back to the window, and a good diffused daylight falling upon the samples. If the standard be found appreciably darker of the two, the value of the sample under examination may be estimated by the number of minims of its liquor which it is necessary to add to an ounce of water to make the tint equal to standard.

Adulterations which include the addition of cheaper colouring matters should always be kept in mind, and will in all probability have given some indication of their presence in the process of making the liquor. Thus Bismarck brown, which gives a yellow tint in very dilute solutions, would be unquestionably brown in the undiluted state, and picric acid would be detected by its intense bitterness. The saffron liquor is but little changed by acids and alkalies ; it is bleached by chlorine and not changed by a drop of tincture of iodine. A dilute yellow liquor of Bismarck brown has similar absence of reaction with the three first reagents, but the iodine develops in it a deep turbid brown coloration. Methyl orange and tropæolin develop a strong red colour with hydrochloric acid.

A common graduated dropping tube answers very well for the above experiments. The accuracy of the minims is not



important so long as the same tube is used for the saffron liquor as has been used for the standard colour.

### CUPRI NITRAS

Nitrate of copper is a chemical of very little pharmaceutical importance. The official tests aim rather at indicating its nature than its purity. The probable impurities are excess of

water, and nitric acid, and the less probable are iron, zinc, lead, and silver.

Twenty-four grains heated to dull redness should leave 8 grains of black oxide of copper, and this on agitation with water and filtration should not impart saline matter to the water, as indicated by a drop of the filtrate evaporating on a slip of glass with an insignificant residue. If it be desired to search for the other metals named above, a little of the nitrate of copper would precipitate white with sulphate of magnesium if lead were present, and white with HCl if it contained silver. The nitrate being totally soluble in liquor of ammonia added in excess, would show the absence of iron; if iron has been thrown down, filter the solution before adding sulphhydrate of ammonium in excess, which will throw down all the copper and any zinc if present. A slight excess of hydrochloric acid being added to these sulphides takes up the zinc but not any copper, and after again filtering, the zinc may be thrown down as a white precipitate on adding ammonia in excess and then sulphhydrate of ammonium; or the filtrate from the copper sulphide may be evaporated and gently ignited, to drive off the ammoniacal salts; any fixed residue would contain zinc, earths, or alkalies, if such were present in the original salt.

### CUPRI SULPHAS

Sulphate of iron is the one common impurity in commercial sulphate of copper. The B.P. test for iron includes the use of chlorine water to oxidise the iron, as ferrous hydrate is soluble to a considerable degree along with the copper in an excess of ammonia, while ferric hydrate is not so; but chlorine water does not keep well and is troublesome to prepare at the moment, so the same object may be more conveniently accomplished by boiling the solution of sulphate of copper with free nitric acid or chlorate of potassium and hydrochloric acid, after which ammonia in excess dissolves the copper but leaves the iron as ferric hydrate.

The U.S.P. gives a more comprehensive test, in acidulating

a 5 per cent. solution of the salt and passing sulphuretted hydrogen through it to saturation, filtering out the sulphide precipitated, and evaporating the filtrate, which should leave no fixed residue if the salt be pure, but will leave sulphates of zinc, iron, alkalies, or earths, if such should have been present in the original sample.

### EXTRACTUM CINCHONÆ LIQUIDUM

This, being a preparation rarely made by the dispensing pharmacist, may be advantageously tested in the following manner. Evaporate fʒij to about ʒj to drive off the spirit, then add ʒj water and ʒss liquor of ammonia '959, and shake with fʒij chloroform in a separating funnel; run off the clear chloroform into a small tared evaporating dish; add to the contents of the separator fʒij more chloroform, shake, subside and run off the clear chloroform as before into the dish, drive off the chloroform at a water-bath temperature till it ceases to lose weight. The contents of the dish should then weigh 5 grains or a little more, but not 6—theoretically 5·46.

### EXTRACTUM NUCIS VOMICÆ

The B.P. test for total alkaloids in this extract can scarcely be improved, either as regards simplicity or efficiency. The only point likely to give trouble to an inexperienced operator is the tendency of the alkaline liquor and chloroform to produce a species of emulsion which prevents the sharp separation of the two liquors which is desirable, and thus involving either a loss of chloroform with some alkaloids or a contamination of it with the aqueous solution and extractive matters. It works better if the solution of the extract in water be filtered before the addition of the carbonate of soda. To effect filtration without appreciable loss, the solution should be passed through a little plug of absorbent cotton rolled not very tightly into a ball about the size of a 5 grain pill, and placed in the throat of a small glass funnel. The solution passes through without difficulty and is clear, and half a drachm of water percolated through



the cotton is sufficient to wash out the absorbed solution. After this filtration the separation of the chloroform is usually quite sharp, and each of the subsequent separations (i.e. when shaken with acid, and when shaken with ammonia and chloroform) are also sharp. In an operation upon 10 grains of extract which yielded by this treatment 1.5 grain of mixed alkaloids, all the residues were again treated, i.e. the plug of cotton with acid, the soda liquor with a second washing of chloroform, the chloroform solution with a second portion of acid, and the ammoniacal liquor with a second portion of chloroform, and the additional portions of alkaloids thus obtained altogether only amounted to 0.1 grain, showing that where the percentage was 16 the B.P. process would indicate 15. This is a result quite close enough to truth to be satisfactory, and as the errors will always be in the same direction, and with good working always of about the same extent, we may take it that 16 per cent. is the official strength, and that this strength is indicated by a yield of 15 per cent. by the official process.

### FERRI ARSENIAS

This is a rather variable compound of ferrous and ferric salt, and is of little medical importance. If it were important to examine it at all, the percentage of arsenium it contains would be the point worthy of consideration ; this, however, is omitted in the official tests. The B.P. tests with the ferrocyanide and ferricyanide of potassium indicate that it is principally ferrous, with a little ferric salt, and the treatment with caustic soda and subsequent addition of nitric acid and silver nitrate indicates that the acid is arsenic and not arsenious. The volumetric examination may be substituted by heating 100 grains in a flask with an ounce of dilute sulphuric acid ; add to this  $3\frac{1}{4}$  grains of potassium bichromate ; the solution will still give a blue precipitate with ferricyanide of potassium, though it will cease to do so on the addition of  $\frac{1}{4}$  or  $\frac{1}{2}$  grain more of the bichromate.

## FERRI CARBONAS SACCHARATA

The faults to be anticipated in this preparation are excess of sugar, the presence of ammonium sulphate (or other alkaline sulphate), and an undue proportion of iron in the ferric condition. The solubility in HCl with effervescence, and the absence, or almost total absence, of a precipitate on the addition of barium chloride, prove the presence of carbonate, and the approximate absence of sulphate. The most important point, the due proportion of iron in the ferrous condition, is officially tested by the volumetric method, the practical equivalent of which may be performed thus :—3ss of the sample dissolved in 3ss dilute sulphuric acid and four grains of potassium bichromate added ; when all is dissolved, a drop of the solution should give a blue precipitate with ferricyanide of potassium, but on the addition of one grain more of the bichromate it should cease to do so. The presence of the sugar in this preparation introduces a possible source of error in this estimation, for though cane sugar is not rapidly attacked by a solution of chromic acid, it is much more readily oxidised after it has been some time in contact with any of the strong mineral acids, especially if heated, and the saccharated carbonate may thus require more chromate for its oxidation than is due to the ferrous carbonate it contains. With the view of avoiding this error the B.P. uses phosphoric acid for the solution of the carbonate of iron, as phosphoric acid has less action on sugar than the hydrochloric or sulphuric acids. The U.S.P. directs hydrochloric acid ; Attfield uses sulphuric. I find no practical difference, so long as heat is not applied, and the contact of the acid is not prolonged. The production of a perfect solution is, however, a slow process, at least in some cases, and the removal of the sugar, which would enable us to use heat and acid freely, is not readily effected without some loss, and some exposure to the air. I have not met with any method by which reliably accurate results can be secured with facility, but probably the small residue, difficult of solution, had better be neglected ; ferrous carbonate being

more readily soluble than ferric oxide, the results will be sufficiently close even when a small portion of the sample remains undissolved. The saccharated carbonate boiled in water does not settle well, nor filter well ; the powder is apt to pass through the paper, and the filtration being slow oxidation is probable ; the addition of an equal bulk of spirit to the boiled solution improves both the subsidence and filtration, but the spirit is as objectionable as the sugar unless it also be washed out.

### FERRI ET AMMONII CITRAS

The impurities to be sought for in this salt are alkaline sulphates introduced through imperfection of the manufacturing process, the substitution, total or partial, of tartaric for citric acid, and the excess or deficiency of ferric oxide. The B.P. process for the estimation of the iron is fairly satisfactory :  $\Theta j$  being heated to redness in a capsule with exposure to air should leave six grains of residue consisting of ferric oxide, free from alkaline reaction on litmus. This test may be further developed by dropping a little strong nitric acid upon the oxide of iron in the capsule, reheating and reweighing. The nitric acid should not cause effervescence, or a fixed alkali is indicated, and the second weighing should correspond with the first. The first burning may have failed to burn off all the carbon, which is easily overlooked from the dark colour of the oxide of iron, and the iron may exist partly as magnetic oxide instead of ferric oxide, both of which errors will be corrected by the treatment with nitric acid.

A sample of the salt which gives no indication of ferrous iron with red prussiate of potassium will, after burning, give copious indication of the same if the burning be not supplemented by the action of nitric acid as above indicated, or some other precaution calculated to convert magnetic into ferric oxide.

If the original salt contains sulphate of sodium the iron residue may contain sulphide if the burning has been insufficient. This would become evident from the evolution of  $H_2S$  on treating it with  $HCl$  or  $H_2SO_4$ , but the treatment with nitric acid



would cause its oxidation, converting all the iron into ferric oxide with the final ignition. In the B.P. process of manufacture, the citrate being prepared from a ferric oxide precipitated by ammonia, there should be no contamination of fixed alkali, but manufacturers often use soda as a precipitant and thus introduce it into the scales. After the burning and treating with nitric acid, the soda may be washed out of the ferric oxide and estimated either by loss of weight of the oxide, or by the evaporation of the washings and weighing the residue.

The absence of tartaric acid is proved by precipitating the iron by heating the salt with excess of caustic potash, filtering and adding acetic acid till there is a freely acid reaction, in which circumstances tartaric acid, if present, would be precipitated as acid tartrate of potassium. This test is rendered more delicate by adding an equal volume of rectified spirit. Some commercial samples of this salt contain notable quantities of ferrous salt, a fault which may result from imperfect oxidation of the iron in the first instance, or may be produced by a reducing action which takes place when there is excess of citric acid. Another defect sometimes experienced is the tendency of a solution of this salt in pure water to undergo rapid deterioration, the solution becoming turbid in a few days, the greater part of the iron being thrown out of solution. I have so far met with no satisfactory explanation of this peculiarity, and it occurs in samples which do not deviate notably from the official characters and tests. It has been suggested that the scales may contain germs of decomposition if, in the manufacturing process, the scaling has been too long delayed. The pharmacopœia does not suggest the testing for sulphates as among the probable impurities, though the process of manufacture naturally involves a considerable chance of the presence of such. As soluble citrates, tartrates, and some other salts retard or prevent the precipitation of barium sulphate, the scales should be heated in water with sufficient HCl to make a clear yellow solution before adding the chloride of barium.

## FERRI ET QUININÆ CITRAS

The iron is not thrown out of this combination by alkaline carbonates nor by caustic ammonia, but is by caustic soda or potash. The quinine is precipitated by either the carbonates or the caustic alkalies ; consequently ammonia gives a white precipitate of quinine in a brown solution of citrate of iron and ammonium, while caustic soda gives a brown precipitate consisting of ferric oxide and quinine in a colourless solution of citrate of sodium. Tannic acid throws down a white tannate of quinine along with a black tannate of iron. When ignited for some time, exposed to the air, it should be converted entirely into ferric oxide. This oxide heated with a small quantity of distilled water and filtered, the filtrate should evaporate without fixed residue, otherwise the scales contain some impurity, probably a salt of the fixed alkalies. Compare citrate of iron and ammonium. The B.P. directs that the yield of quinine from 50 grains shall be  $7\frac{1}{2}$  grains ; it is most conveniently estimated by putting the given quantity of the scales into a separator with an ounce of water, adding ammonia sufficient to precipitate the quinine, and then successive portions of chloroform, two or three fluid drachms each time, giving time for subsidence, and running off each time only so much as can be separated quite free of aqueous liquor, the chloroform liquors being run direct into a small tared evaporating dish. This is more convenient than using ether, the decantation of the ether from the surface of the water, which is not so completely effected without admixture, or the running off of the aqueous liquor involving loss by the repeated transference from one vessel to another.

## FERRI PEROXIDUM HYDRATUM

The commercial ferric hydrate may contain ferrous hydrate or carbonate ; the latter will produce effervescence with HCl ; either will produce a solution yielding a blue precipitate with ferricyanide of potassium. Anhydrous ferric oxide should be

guarded against, as it is much less soluble. The only other probable impurity is sodium sulphate or chloride from imperfect washing.

### FERRI PHOSPHAS

The impurities of this salt are almost confined to excess of the ferric phosphate and oxide. Excessive oxidation will be indicated by 30 grains dissolved in f3ss dilute hydrochloric acid and treated with 4 grains of potassium bichromate failing to give a blue precipitate with ferricyanide of potassium. Four and a half grains should have the effect of completing the oxidation of the iron, and consequently of producing with the ferricyanide only a clear, dark blue-green coloration ; otherwise more than the usual proportion of iron is in the ferrous condition.

When treated with tartaric acid and subsequently with ammonia in excess, tartrate of iron and phosphate of ammonium are produced, and the presence of phosphoric acid is subsequently indicated by the addition of ammonio-sulphate of magnesium producing an abundant precipitate of minute crystals of ammonio-phosphate of magnesium.

The B.P. test with copper foil and HCl is Reinsch's test for arsenic ; it is intended to indicate that arseniate of iron has not been substituted for the phosphate, which it in some slight degree resembles.

### FERRI SULPHAS

The only impurities to be anticipated in the recrystallised ferrous sulphate of pharmacy are ferric sulphate and oxide. The aqueous solution is often turbid from a little free oxide, and it is almost impossible to obtain a precipitate approaching whiteness with ferrocyanide of potassium, though it is theoretically possible and apparently demanded by the B.P. The official tests also require that it shall give no precipitate with  $H_2S$ , which may be the case if the salt be quite pure, though a little ferric sulphate would cause precipitation of white sulphur, or a little free oxide of iron would cause a black pre-



cipitate of sulphide of iron. If a little sulphuric acid be added before testing with  $H_2S$  and a black precipitate is formed, copper would probably be the impurity present, though this is a contingency scarcely to be anticipated. The volumetric test is unnecessary, but if it be desired to test its capacity for oxidation 84 grains dissolved in an ounce of water with  $\text{3ij}$  dilute sulphuric acid and 14 grains of bichromate of potassium should still be capable of giving a blue precipitate with ferricyanide of potassium, but on the addition of one grain more of the bichromate it should only be capable of giving a dark green colour without precipitation. The failure to stand this test might result from excess of water, or less probably from the presence of sulphate of sodium, magnesium, or aluminium.

#### FERRI SULPHAS EXSICCATA

The pharmacopœia allows this to be short of its theoretical capacity for oxygen to the extent of about  $2\frac{1}{2}$  per cent., which may result from excess of water, or from oxidation in the process of drying. The deficiency will generally be found greater than this, and results from both causes, and both of these increase by keeping. It may be considered in fair condition if 20 grains in 1 ounce of water with 2 drachms of dilute sulphuric acid and 5 grains of potassium bichromate yields a blue precipitate with ferricyanide of potassium, but ceases to yield such when one grain more of the bichromate is added ; the reaction should then be a dark green coloration without any precipitate.

#### FERRI SULPHAS GRANULATA

Except as regards the size of the crystals, this is practically the same as the ferrous sulphate made in the usual way. It is reputed to have less tendency to absorb oxygen than that crystallised without spirit ; but of this I have not seen evidence. The B.P. requires that 41.7 grains of the granular sulphate of iron shall require 7.37 grains of potassium bichromate for its complete oxidation, and as it states that 42.1 of the ordinary crystals

require the same, it is apparently assumed that the granules will have their full theoretic value and the crystals will contain 0.4 parts in the 42.1 of unoxidisable matter, presumably adhering water. In actual practice neither of these results can be looked for as absolute. The comparative insolubility of proto-sulphate of iron in spirit, advantage of which is taken in the official process to throw down the salt in a granular form without at the same time precipitating the ferric sulphate or sulphuric acid, theoretically affords the means of procuring a pure salt ; but the commercial granular sulphate of iron is probably rarely produced by the official method, and is certainly not as a rule free from oxidation.

### FERRUM REDACTUM

The pharmacopœia states that this contains a variable amount of oxide of iron, but the test limits it to 50 per cent. or under. The maceration of 10 grains of reduced iron in water with 50 grains of iodine and 50 of potassium iodide should dissolve all the metallic iron and leave all the oxide. The weight of the insoluble matter exceeding 5 grains would indicate an insufficient reduction of iron in the process of making, or subsequent oxidation in keeping.

The oxide left after treatment with iodine should be soluble in HCl, but as the whole of the reduced iron should be so soluble it is better to take a fresh sample, and note at the same time the absence of odour of  $H_2S$  while solution is going on, as the preparation may contain sulphide of iron if made from a ferric oxide thrown down from ferric sulphate and imperfectly washed. The U.S.P. requires the presence of at least 80 per cent. of metallic iron ; that found in English pharmacies frequently contains less than 50 per cent. A strong heat which is favourable to a high percentage of metallic iron renders it more gritty and granular in texture, and less subject to oxidation by keeping. In a smooth-feeling dark sample we may anticipate a deficiency of metallic iron ; in a rough grey one, probably an excess over the official requirement. The B.P. process for estimating the

metallic iron, i.e. the weighing of the residue insoluble in a solution of iodine, is more complicated than necessary. Ten grains of the sample put into a test tube with two drachms of water and 22 grains (theoretically, 22·3) of iodine will speedily set up a brisk reaction which, when it has subsided, may be further aided and completed in a few minutes by the application of heat, producing a solution of iodide of iron free from brown colour, and containing the black oxide as an insoluble residue. The presence of the oxide does not make any difficulty in observing the colour of the solution, and the absence of a brown tint shows that the 10 grains contained enough iron (5 grains) to combine with all the iodine used. The iodide of potassium used in the B.P. test is not necessary. Though iodine is very sparingly soluble in water, it is enough to start the reaction, and the iodine is freely soluble in the solution of iodide of iron thus produced.

### FERRUM TARTARATUM

A solution of this salt should not contain any ferrous oxide, the freedom from which is directed by B.P. to be proved by the absence of a blue precipitate on adding ferricyanide of potassium to a solution acidulated with HCl ; but, as the HCl causes a brown precipitation of the iron at first, it is better to add it freely and boil till the iron is converted into pale yellow chloride, and then to dilute with water and test with the ferricyanide. The presence of potassium is to be proved by getting rid of the iron by precipitating with caustic soda and heat ; the filtrate from the iron precipitate contains tartrates of sodium and potassium with some excess of soda ; when this is rendered acid with acetic acid, the potassium and tartaric acid go down as acid tartrate of potassium.

In this precipitation care should be taken to have the solution cold, and not much diluted, or the precipitate is not promptly formed ; it may be expedited, if necessary, by adding an equal volume of rectified spirit.

In estimating the ferric oxide by incineration, 50 grains should yield 15 grains of ferric oxide, but to get this result



correctly requires attention to two or three points. The iron, though in the ferric condition before burning, is partially reduced by the tartaric acid at a red heat, and oxidation is much retarded by the presence of the potassium carbonate, resulting from the charring of the tartrate; it may also contain carbon, and before it can be weighed as oxide of iron it requires washing to remove the alkali and reburning to complete the oxidation of the carbon and iron; this is most readily secured by putting a little strong nitric acid upon the iron residue before the final burning.

### GLYCERINUM

The glycerines of commerce which answer to the description of being 'clear, colourless fluids' are generally free from metallic and earthy impurities, and, if free from odour and acrid taste, may be safely regarded as fit for all external purposes; but, for internal use, it is important to prove the absence of lead by there being no coloration with sulphuretted hydrogen or sulphhydrate of ammonium. A black or brown colour with this latter reagent would probably be caused by lead, though copper or iron might also be the cause of this reaction.<sup>1</sup> Glycerine, if distilled without due precautions, suffers partial decomposition, evolving acrid vapours of acrolein, and introducing this body as an impurity into the distillate. Acrolein produces a white precipitate with nitrate of silver, and, under the influence of heat or light, reduces some of the silver to the metallic state. Good glycerine produces no precipitate with solution of nitrate of silver, but even the highest brands are more or less tinted yellow, pink or purple-brown, with this reagent when exposed some time to daylight.

The freedom from coloration when mixed with an equal volume of strong sulphuric acid indicates the absence of sugar or dextrine.

Another test for saccharine bodies is to heat a sample in a

<sup>1</sup> Arsenic has also been found in some of the inferior glycerines recovered from the waste liquors of soap manufacture. (See 'Chemist & Druggist,' xxxv. p. 368, and Feb. 22, 1890; 'Pharm. Journ.' xiv. p. 865; also 'Year Book of Pharmacy,' 1889.)

platinum dish or spoon till the glycerine is driven off; the residue will be a porous char if sugar be present, but only a black stain if the glycerine be pure, and this stain should burn off at a red heat, leaving a scarcely visible ash.

Butyric acid is sometimes present in glycerine and may be detected by its rancid odour if the sample be warmed with a little dilute sulphuric acid.

### GOSSYPIUM

The cotton-wool of the B.P. is an 'absorbent cotton' produced by treating crude cotton with alkali and other chemicals to remove the fatty matter, &c. The processes used for the removal of fat and colour may introduce other impurities, hence the necessity for proving its freedom from acid or alkali by the use of test paper, and the burning off of a given weight. The ash of 100 grains should be less than 1 grain, and should be nearly white. Pure cellulose should, theoretically, leave no ash, but long-continued action of hydrochloric and hydrofluoric acids fails to remove it entirely, and the natural ash is sometimes added to by the use of blue to cover the natural yellow tint of the cotton.

### GUAIACI LIGNUM

The treatment of guaiacum chips with nitric acid is intended to indicate by the production of a green colour that the chips are taken from that part of the stem which contains the resin in greatest abundance; the external pale yellow sap-wood contains so little resin as not to produce a green colour under the action of oxidising agents. A weak solution of chlorinated lime is a better reagent than nitric acid for developing the blue-green colour.

### HYDRARGYRI IODIDUM RUBRUM

The change of colour from red to yellow on heating is not the result of decomposition, and though the yellow colour may continue some time after it is cold the red is immediately restored by the slightest friction.

When digested with solution of soda (caustic) red oxide of mercury is formed, while the double iodide of sodium and mercury remains in solution ; from this the iodine may be set free by the addition of nitric acid, and its presence shown by its yellow-brown colour and the blue-black precipitate it forms with solution of starch. These characters are given chiefly to indicate that the sample under examination is really the iodide of mercury, but the change of colour by heat would reveal the presence of red sulphide of mercury or other red powders with which it might be mixed, as in that case a pure lemon colour would not be produced. A little of the powder heated should be entirely volatilised below red heat (excluding such fixed impurities as might possibly be present). A drachm of the iodide being shaken with two drachms of water and thrown on a filter, the filtrate should evaporate with only a trace of residue. Treated in the same way with rectified spirit on evaporating fʒj of the filtrate the residue should not exceed 0·3 grain. Four grains of the sample should be totally soluble in fʒv of ether. These solubilities would prove the absence of all probable impurities.

#### HYDRARGYRI OXIDUM FLAVUM

The entire solubility of this in HCl proves the absence of the grey oxide of mercury, metallic mercury, or calomel. The solution of perchloride of mercury formed throws down ammoniated mercury on the addition of ammonia. The entire volatility at a low red heat proves the absence of oxide of iron and other non-volatile impurities. There are no probable impurities which would not be detected by these tests. If shaken with distilled water and thrown upon a filter the filtrate should evaporate without appreciable residue ; most of the substances capable of being detected by this test would also be indicated by being left on ignition.

#### HYDRARGYRUM OXIDUM RUBRUM

This is, chemically, the same as the yellow oxide ; its darker colour is due to its less finely divided condition. It should



stand the same tests as the yellow oxide, and as it may contain nitrate from the mode of its production, the test of heating should be conducted in a test tube, and the absence of yellow nitrous fumes noted. It is said to be sometimes contaminated with potassium nitrate, which would of course remain after the volatilisation of the mercurial compounds. The U.S.P. attempts to distinguish between the yellow and red oxides of mercury by the comparative facility with which a solution of oxalic acid converts the former into white oxalate of mercury.

### HYDRARGYRI PERCHLORIDUM

This salt rarely contains any appreciable impurity. The B.P. tests are chiefly directed to prove its nature, but its subliming without decomposition and without residue excludes many impurities—such as nitrate of mercury, chloride of potassium or sodium. The U.S.P. adds a test for arsenical compounds, impurities not at all probable, but which the sublimation test would fail to indicate. The presence of sulphate would be detected by  $\text{BaCl}_2$ , and if ammonium chloride were present the addition of caustic potash would give a white instead of a yellow precipitate, ammoniated mercury instead of yellow oxide.

### HYDRARGYRI PERSULPHAS

This salt has scarcely a place in pharmacy except as a step in the production of other mercurials. Its complete volatilisation without decomposition is the only indication of purity given. The salt is readily identified by its decomposition when thrown into a bulk of water—the basic yellow sulphate being precipitated while an acid sulphate remains in solution.

A general comprehensive test is to treat it with excess of barium carbonate in distilled water, and filter; the filtrate should be nearly pure water, and evaporate with an insignificant residue. The operation may be advantageously performed thus: Six grains of the sulphate boiled in a test-tube with three drachms of water and allowed a minute to subside; the clear, or nearly

clear, solution of acid sulphate of mercury decanted into another tube, leaving the bulk of the yellow insoluble oxy-sulphate behind, adding to the decanted portion 6 grains or more of precipitated barium carbonate, and again boiling, filtering the liquor into a small dish or watch-glass and evaporating to dryness; the residue should be less than 0.05.

The native carbonate of barium is not suited for this use, as it is much less readily attacked by sulphates, and the precipitated carbonate should be so free from soluble impurities that distilled water shaken with it leaves an insignificant residue when a drop is evaporated on a slip of glass. An excess of the carbonate is advantageous, as it expedites the complete decomposition of the sulphate.

#### HYDRARGYRI SUBCHLORIDUM

The most probable and most important impurity in calomel is perchloride of mercury, which may be detected by its taste and by its ready solubility in ether or spirit, with which the calomel may be washed. Warm ether is directed to be used for this purpose; hot water or warm spirit is a much more convenient, and quite sufficiently active solvent of the perchloride, though subsidence takes place much more rapidly in ether. The treatment of calomel with caustic potash or lime converts it into black oxide of mercury and chloride of the alkaline metal.

#### HYDRARGYRUM

Mercury, if contaminated with other metals, loses some of its fluidity, and the globules are less perfectly spherical. The tendency to tarnish is also an indication of impurity. As a final and conclusive test, its complete volatilisation below red heat is simple and satisfactory, other bodies capable of being volatilised at such a temperature not being miscible with the metal in its fluid state.

## HYDRARGYRUM AMMONIATUM

White precipitate varies in composition from little variations in the mode of its manufacture ; one variety containing less mercury is fusible before it volatilises, and the B.P. requires that ammoniated mercury shall volatilise without fusion to exclude the fusible variety from pharmaceutical use. The reduction of 100 grains to the metallic state by boiling with stannous chloride is more troublesome, but if it yields 77·5 grains of mercury it is more conclusive of the correctness of the sample.

## HYDRARGYRUM CUM CRETA

The serious impurity to which grey powder is liable is oxide of mercury. By continued action of air black oxide of mercury is formed, and this, especially under the influence of heat or light, is converted into red oxide and metallic mercury. The presence of the red oxide is detected by its solubility in HCl and subsequent precipitation by stannous chloride, or by the solution depositing metallic mercury upon a copper coin. The black oxide of mercury is converted into calomel by the HCl, and is left mixed with the unoxidised mercury, upon which the acid has no action. If the grey powder be treated with acetic acid, both the oxides of mercury are dissolved as well as the chalk, leaving only the finely divided metal behind. The addition of HCl to the acetic solution will then precipitate as calomel any mercury which had existed as black oxide, and the solution will retain that which was peroxidised. This enables the estimation of the mercury in each of its three states to be completed, the solution after separation of the precipitated calomel being treated either with stannous chloride to reduce metallic mercury, or with  $H_2S$  to precipitate it as sulphide.

This examination is instructive and interesting, but to most pharmacists it will be enough to know that the solution obtained by treating 3j grey powder with dilute hydrochloric acid



in slight excess and filtering should not precipitate red with a single drop of solution of iodide of potassium.

In all these examinations of grey powder it is desirable to operate quickly and without heat, as mercuric compounds are readily converted into mercurous by the action of finely divided mercury. Deficiency of mercury is a contingency not to be overlooked, and may be most readily detected by the action of iodine. The operation may be performed as follows: Rub 30 grains of grey powder in a mortar with 20 grains of iodide of potassium and 12 grains of iodine, adding a few drops of water to reduce it to a pulpy condition. The colour should shortly become pale grey with only a tint of yellow. The continuance of a brown colour would indicate deficiency of mercury or considerable oxidation. The 30 grains of grey powder should contain 10 of mercury, which would combine with 12·7 of iodine to form red iodide of mercury, and this, in combination with the iodide of potassium, produces the pale yellow double iodide of mercury and potassium. The use of 12 grains instead of 12·7 of iodine allows for a little oxidation and small errors.

### IODOFORMUM

A drachm of iodoform being shaken with an ounce of water and thrown upon a filter, the filtrate on evaporation should leave only a trace of residue (under 0·1 grain). Treated in the same way with rectified spirit (at about 60° F.), the filtrate on evaporation should leave *about* 1·2 grains. As iodoform is slowly volatile, this is only an approximate figure, and a drachm treated with ether should entirely dissolve in a fluid ounce. These solubilities will exclude the majority of probable and improbable impurities. A further confirmation is obtained by spreading a grain over a slip of glass by the aid of a drop or two of water or spirit, and exposing it at about water-bath temperature, when it will slowly but entirely volatilise without decomposition. At higher temperatures it is decomposed, but should entirely burn away at a red heat.

## IODUM

The resublimed iodine of pharmacy rarely contains any impurity. The commercial quality contains a small quantity of moisture and a trace of impurity not volatilised. It may also contain cyanogen, chlorine, or bromine in combination, which, being volatile, are not indicated by any fixed residue. Cyanogen is a serious but a rare contamination. It is indicated by the B.P. test of volatilisation at a low heat ; cyanide of iodine being more volatile than iodine, it comes over first, and condenses in slender colourless prisms with a pungent odour. Chlorine in combination increases the solubility of iodine, and may be suspected if the sample produces a strongly brown solution when shaken up with water. A drachm of iodine shaken with an ounce of cold water and filtered, the filtrate should be of a pale brownish-yellow colour, and completely decolorised by the addition of half a grain of hyposulphite of soda.

## JALAPÆ RESINA

The official jalap resin is a mixture of several resinous bodies, all of which are soluble in rectified spirit, and none of which is soluble in oil of turpentine. The powdered resin, being treated with warm water and filtered, the filtrate should leave only a trace of residue on evaporation. Commercially it is rarely without this trace of residue, consisting of deliquescent extractive matter of the jalap root which is not readily completely washed out, but so long as it is only a trace it is of no moment.

Resinous matter soluble in oil of turpentine would be a sophistication not to be anticipated in any sample from a good source. The B.P. also requires that not more than 10 per cent. should be soluble in ether, a character which discriminates between it and the resin of scammony, which is totally and freely soluble in ether.

## LIQUOR AMMONIÆ FORTIOR

The solutions of ammonia, as found in pharmacy, rarely contain any impurity affecting their medical use, or their application to pharmaceutical operations. The official test with lime-water is intended to show its freedom from carbonates—oxalate of ammonium refers to lime—sulphydrate of ammonium to metals—and ammonio-sulphate of copper to sulphides.

The strength of the solutions is a point in which deviation from standard is more likely to occur. The stronger solution is calculated to contain 32·5 per cent. of ammonia gas by weight, and on dilution by volume, with two parts of water, produces within a small fraction a ten per cent. solution. The operation of diluting with two volumes of water is the simplest possible, but the relative strengths of the two liquors, as given in B.P. characters and tests, are obscured by the complicated relationships between a fluid drachm (=54·68 grains by measure), multiplied by sp. gr. 0·891, of a 32·5 per cent. solution, diluted with two fluid drachms of water, producing 3 fluid drachms of a sp. gr. 0·959 containing 10 per cent. of ammonia.

It is customary to indicate the strengths of the ammoniacal liquors by their density, and though authorities differ as to the relationships which subsist between densities and percentages, it is enough for the pharmacist if his densities agree with those given in the B.P.

A fluid drachm of the strong liquor should neutralise 58·6 grains of oxalic acid, but as there is loss of ammonia by evaporation while operating, it is best to dissolve 60 grains of oxalic acid in two ounces of distilled water, colour it with litmus, and pour into it in full stream a fluid drachm of the ammonia, rinsing the drachm measure with water, and adding this to the acid solution, which should continue to retain its pink tint, but become about neutral with one drop more of the ammonia, or alkaline with two drops ; this is as close a result as can be looked for with pharmaceutical measures, and closer than would be obtained by weighing 52·3 grains of liquor of



ammonia, as suggested in the B.P. unless special appliances and precautions were adopted to prevent loss, precautions not so readily carried out at the dispensing counter.

### LIQUOR AMMONII ACETATIS

The B.P. gives no test for this but its sp. gr. and neutrality ; the point of neutrality is not sharply indicated by the colour of litmus, and to insure any approach to correctness, it is necessary to heat the solution to drive off the carbonic acid before testing, as the latter acid, while retained in solution along with the carbonate of ammonium, gives the reaction of a neutral salt long before all the carbonate has been converted into acetate ; it may also be noted that heat drives off ammonia from a neutral or even from an acid salt, so that you may have a strongly acid solution of acetate of ammonium, while hot, smelling strongly of ammonia. For pharmaceutical use, a little excess of acetic acid is usually less objectionable than an excess of carbonate of ammonium, as, for example, when it is to be given in conjunction with a solution of morphine, the alkaline condition would cause the precipitation of the alkaloid ; but, on the other hand, if it is to be given in mixture with spirit of nitrous ether and iodide of potassium, the alkaline condition would be the least objectionable.

It is best in making the solution by the official method to add the final quantity of acetic acid by small portions, till a drop of the solution taken up with the stirring-rod and let fall upon a drop of neutral solution of acetate of lead causes no white precipitate.

It is most convenient for this test to have some black surface, spotted with drops of the lead solution, so that repeated trials may be conveniently made while the process proceeds ; the diminution in the density of the precipitate indicates the approach to neutrality, and when it becomes very faint shows the propriety of adding the acetic acid with caution.

It is important that the lead solution be neutral ; the liquor plumbi subacetatis is not suitable, as it gives a white precipitate

with free carbonic acid, if the acetic acid be not in considerable excess ; but the neutral solution produced by dissolving crystallised acetate of lead in distilled water, and if necessary filtering, gives a white precipitate, with a very slight trace of carbonate of ammonium, but none with free carbonic acid. It is, of course, useless to apply any test for neutrality till the last fragment of the ammonium carbonate is in solution, but after this is effected the effervescence may be disregarded as it in no way affects the application of this test.

If the process of making acetate of ammonium be reversed, as is sometimes done, that is, adding carbonate of ammonium to acetic acid of a suitable strength till neutrality be obtained, the lead test requires a little modification. The neutral acetate of lead in that case would give no indication of the approach to neutrality, so the solution of the subacetate of lead may be used till it gives a precipitate, which it will do before the acid is completely neutralised, and then the additions of carbonate of ammonium should be made with caution till the first appearance of a faint precipitate when testing with the neutral acetate. As a further precaution, a portion of the acetate of ammonium solution may be taken from the bulk when neutrality is approaching, and returned again when the bulk gives the first faint indication of alkalinity.

A drop of the solution of acetate of ammonium on a slip of glass should entirely evaporate below  $212^{\circ}$  F., with at least a partial dissociation of its constituents.

The old method of making solution of acetate of ammonium was by neutralising dilute acetic acid with carbonate of ammonium, and an impression was common that by taking an acid eight times the strength, and neutralising it with carbonate of ammonium, a solution was obtained eight times the official strength. The present mode of procedure is calculated to dispel that misapprehension, and a testing of the gravity gives all requisite security on this point.

The density of the weaker solution should be 1.013 and that of the stronger 1.070.<sup>1</sup>

<sup>1</sup> There is apparently some error in the B.P., as the densities indicated,

Ammonia and several of its salts have considerable solvent powers, and if kept long in bottles made of lead glass, are apt to dissolve lead out of the glass, giving the inside of the bottle a milky appearance. The quantity of lead thus abstracted is usually too small to be detected by sulphhydrate of ammonium unless the contact of the ammoniacal solution has been very long continued. Common green glass contains no lead, and is not thus attacked. Though the B.P. directs the store bottles to be free from lead, no test is given to indicate that the solutions of ammoniacal salts are free from this impurity. Acetic and citric acids and liquor of ammonia are all directed to be tested with  $\text{H}_2\text{S}$ , and there is the same ground for requiring the application of this test to the solutions of the ammoniacal salts unless they have been produced from tested chemicals. A trace of lead sufficient to produce a pale brown coloration with  $\text{H}_2\text{S}$ , or sulphhydrate of ammonium, is not improbable and not to be overlooked.

### LIQUOR ANTIMONII CHLORIDI

The yellowish red colour which this solution usually has, is dependent upon ferric chloride which it contains as an impurity; a solution of pure chloride of antimony being free from colour. The precipitate that falls when liquor of chloride of antimony is diluted with water, is an oxychloride ( $2\text{SbO}_3 + \text{SbCl}_3$ ), hydrochloric acid and chloride of antimony remaining in solution. Many other salts of antimony behave in a similar way, but the presence of tartaric acid prevents this precipitation, and does not interfere with the precipitation of antimony as a sulphide; it is consequently used in the B.P. testing to insure that all the precipitate capable of being produced by  $\text{H}_2\text{S}$  is sulphide of antimony, and not a mixture of the sulphide with oxychloride which might result if the same operation were attempted without the presence of tartaric acid. The orange colour of the sulphide

i.e. 1.022 and 1.073 respectively, are not produced by following the official instructions.



obtained by this test proves the absence of copper, lead, bismuth, and some other metals, which throw down black sulphides in the presence of acids, but does not guard against tin and arsenic, the light yellow sulphides of which would not be seen in the presence of the orange-coloured sulphide of antimony, nor does it prove the absence of iron and zinc, the sulphides of which are only precipitated in the absence of free acid. The precipitated sulphide of antimony usually contains some uncombined sulphur ; this is sure to be the case if the chloride contained ferric chloride. The B.P. requirement, that a fluid drachm of the liquor shall yield a precipitate weighing *about* 22 grains, is evidently intended to allow a little latitude ; the test is not of great importance.

### LIQUOR ARSENICALIS

The addition of HCl or some other acid, sufficient to give the solution an acid reaction, is necessary before testing it with  $H_2S$ , as the sulphide of arsenium is soluble in alkaline liquors. In the volumetric estimation with iodine, the  $As_2O_3$  is converted into arsenic acid  $As_2O_5$ , and the test is only given on theoretical grounds ; anyone competent to undertake it would rather make his own liquor arsenicalis and save the necessity of the analysis.

### LIQUOR ARSENII ET HYDRARGYRI IODIDI

Sulphuretted hydrogen throws down a black precipitate consisting of the sulphides of arsenium ( $As_2S_3$ ) and mercury ; on the subsequent addition of nitric acid, the former only is decomposed, oxidised, and dissolved, the sulphide of mercury being left as a black precipitate, and when this is separated by filtration and the solution again treated copiously with  $H_2S$ , or sparingly with sulphhydrate of ammonium, sulphide of arsenium will be thrown down as a yellow precipitate which may now be  $As_2S_5$  with some uncombined sulphur, the nature of the precipitate being partly dependent upon the degree of heat and quantity of nitric acid used. The more nitric acid there is

present in the solution, the more  $H_2S$  is required, and the more sulphur is liberated from it; hence the B.P. instruction to use sulphydrate of ammonium which neutralises the free acid; but as the sulphides of arsenium are freely soluble in excess of the sulphydrate of ammonium, it is necessary to have regard to the B.P. instruction to add this reagent *gradually*. The most probable defect of this preparation is the deficiency of iodide of arsenium, the commercial iodide frequently containing uncombined metallic arsenium or its suboxide. If it were necessary to estimate the arsenium and mercury, iodide of potassium and caustic potash being added to produce a clear alkaline liquor, the addition of sulphydrate of ammonium would precipitate sulphide of mercury, which may be filtered off, washed, dried, and weighed; and the filtrate will contain the arsenium, which may be thrown down as sulphide on adding  $HCl$ . The sulphide of arsenium is not to be depended upon as of constant composition under ordinary circumstances, and especially so when thrown down from solution in sulphydrate of ammonium; the precautions to be taken in estimating arsenium as sulphide with accuracy, will be found in most treatises on analysis, and are scarcely such as will be undertaken by a dispenser in the examination of his arsenical liquors.

#### LIQUOR BISMUTHI ET AMMONII CITRATIS

The most probable faults in this preparation are too much ammonia or too little bismuth. If the ammonia is not in too great excess, a small addition of acid will throw down citrate of bismuth. The proportion of bismuth is conveniently estimated as the B.P. directs. The probable impurities are those of bismuth, which see (p. 60).

#### LIQUOR CALCII CHLORIDI

Not much subject to impurity. It may contain traces of  $Mg$ ,  $Al$  or  $Fe$ , any of which would be precipitated by the addition of lime water.

## LIQUOR CALCIS

This should be tested as regards its strength ; it may be considered satisfactory so long as a fluid ounce more than neutralises a grain of oxalic acid. The B.P. volumetric test requires that 10 fluid ounces shall neutralise 11·3 grains of oxalic acid. The most convenient mode of working is to colour 2 fluid ounces of lime water with a few drops of tincture of litmus and add a solution of 2 grains of oxalic acid, when a white precipitate of oxalate of calcium falls in the solution, which should retain its blue colour. Should the tint be red, the lime is deficient. This is all that the pharmacist usually requires, but if it be desired to estimate the extent of deficiency, more of the lime water may be added gradually till the blue colour is restored, when it will be seen how much of the sample in hand is equal to two ounces of good quality.

## LIQUOR CALCIS CHLORINATÆ

The B.P. allows more latitude as regards the strength of this preparation than seems desirable to allow in any case where standardising is ordered, any strength from 2 to 3 per cent. of available chlorine being within the official limits. The B.P. volumetric test reduced to dispensing weights and measures stands thus : 84 minims of the liquor with ℥j iodide of potassium in 4 ounces of water, when acidulated with fʒij hydrochloric acid, should form a brown solution, which requires for its decolorisation at least 11·16 grains of hyposulphite of soda, and when good and fresh may require as much as 16·74 grains to produce this effect.

A reasonable limit, within the official bounds, would be to require a fluid drachm with ℥j of KI in 4 oz. water, acidulated with fluid fʒij of hydrochloric acid, to retain a yellow-brown colour on adding 10 grains of hyposulphite of soda, but to lose its colour on adding two grains more of the hyposulphite.



## LIQUOR CALCIS SACCHARATUS

The only test necessary to apply to this solution is its capability of neutralising oxalic acid. A fluid ounce coloured blue with litmus should retain its blue tint on the addition of 15 grains of oxalic acid previously dissolved in water, but should become neutral or faintly acid on adding another grain.

## LIQUOR CHLORI

This solution is so deficient in stability that, unless newly made, it will almost certainly contain less than its legitimate quantity of free chlorine.

To a fluid ounce of the chlorine liquor add ℥j iodide of potassium, dissolved in an ounce of water; the brown solution thus produced should not be entirely decolorised by 18 grains of hyposulphite of soda, but an additional grain should render it colourless. If it be desired to estimate the degree of deficiency in a weak sample which has been entirely decolorised by 18 grains of hypo, add by degrees more of the chlorine solution, and the first faint yellow tint which becomes permanent will indicate that so much as has been used of the sample in hand is equal to a fluid ounce of a solution of fair quality.

## LIQUOR FERRI ACETATIS FORTIOR

The absence of a blue precipitate with ferricyanide of potassium is required to prove that all the iron is in the ferric condition. The only other probable impurity is sulphate of ammonium, which may be present from imperfect washing of the ferric hydrate, and would be detected in the usual way by barium chloride. Deficiency of iron, which by the B.P. is ascertained by precipitation with ammonia, washing, drying and igniting the precipitate, is, at least, equally probable. A fluid drachm should thus yield 5·7 grains. A simpler operation is to evaporate a fluid drachm in a small capsule and ignite the

residue, which should then consist of not less than  $5\frac{1}{2}$  grains of ferric oxide. On treating this with a few drops of water, filtering and evaporating the filtrate, a mere trace of saline matter should remain.

### LIQUOR FERRI DIALYSATUS

The total absence of chlorine required by the B.P. is a theoretical perfection scarcely to be looked for in practice. The liquor should contain 5 per cent. of ferric oxide, the official method of determining which is by precipitation with ammonia, washing and igniting the precipitate ; the yield from 100 grains of the liquor being 5 grains of the oxide.

A simpler operation is to evaporate f3ij of the liquor in a capsule and ignite the residue, which should weigh  $5\frac{1}{2}$  grains and yield no soluble matter to a few drops of water.

### LIQUOR FERRI PERCHLORIDI FORTIOR

This liquor may contain  $\text{FeO}$ ,  $\text{Fe}_2\text{O}_3$ , As, Na, Ca, free hydrochloric acid, or nitrous fumes. The last constituted a common impurity when the present process was first introduced, but manufacturers do not now commonly follow this method, and it is rarely found ; its presence is to be detected by mixing f3j of the liquor with f3j strong sulphuric acid, and when cool dropping in a crystal of protosulphate of iron, the blackening of the surface of which would indicate the presence of a nitric compound. Arsenium is not a probable impurity, but if present may be detected by the B.P. method ; f3j of the solution with f3j of water, boiled in a test-tube with a piece of clean copper wire, the wire rinsed with water and dried by heat without friction, will have a black coating containing arsenium if such were present. On heating the wire in a clean dry test-tube the arsenium becomes oxidised, volatilised, and condensed upon the cooler part of the tube in the form of a white sublimate of arsenious acid, consisting of minute crystals in octahedral and allied forms. The presence of ferrous iron may be detected by

ferricyanide of potassium, producing a blue precipitate in a little of the liquor diluted with water. If ferric salts only are present, a clear green solution should be produced. To detect  $\text{Fe}_2\text{O}_3$  which is a common impurity but not a serious one, take f3ss of the liquor in two ounces of water and boil in a flask, when oxychloride, if present, will be thrown down as a brown precipitate. This precipitation only takes place in dilute solutions. A rich brown colour in the strong liquor may be taken as an indication of the presence of ferric oxide, and if this becomes paler on the addition of a little strong hydrochloric acid, this is confirmed without the application of the test by dilution and ebullition. On the other hand, if free hydrochloric acid be present, no precipitate will be formed on boiling, and if a drop of weak solution of ammonia be added to the dilute solution, the precipitate at first formed redissolves on agitation. Processes of manufacture other than the official method might give fixed alkaline or earthy salts as impurities, which would be detected by precipitating all the iron with an excess of ammonia, filtering and evaporating the solution, when the residue of chloride of ammonium should be entirely volatile below red heat.

The precipitation of ferric oxide as a means of estimating the ferric chloride may be supplanted by precipitation as ferrocyanide in the following way :—611 parts of yellow prussiate combine with 160 of  $\text{Fe}_2\text{O}_3$  to make prussian blue ; consequently if a fluid drachm should yield between 15 and 16 grains of the oxide it should require between 57 and 61 grains of the yellow prussiate for complete precipitation. Take 61 grains of yellow prussiate, dissolve in sufficient water to make a fluid ounce of solution, add f3vijss of this to f3j of strong solution of perchloride of iron previously diluted with f3vij of water ; mix thoroughly, take a drop on blotting paper, and as the comparatively colourless solution spreads in the paper, drop a little of the ferrocyanide of potassium solution on the paper so near that the two drops run together ; a further precipitation of blue should take place in the paper where the two drops join, or the iron is deficient. If now the remaining



f5ss of the prussiate solution be added, testing on the paper in the same way, no further development of blue should take place, or the iron is in excess. There is usually no difficulty in getting a clear margin to the drop of blue on the blotting-paper, but the use of spirit (methylated) to dilute the iron liquor insures the precipitate separating promptly and perfectly.

### LIQUOR FERRI PERNITRATIS

One fluid drachm evaporated to dryness in a capsule and ignited, should leave a residue weighing not less than  $2\frac{1}{2}$  grains and not more than  $2\frac{3}{4}$ , and consisting of ferric oxide yielding little or no soluble matter to water.

### LIQUOR FERRI PERSULPHATIS

This is scarcely used in pharmacy except as a source of ferric oxide in the production of the scaled salts ; it should be free from ferrous salt as indicated by the absence of blue precipitate with ferricyanide of potassium, and its strength should be accurate, as indicated by its gravity being not less than 1.44 and not more than 1.444. Or by the precipitation method as described under solution of perchloride of iron. A fluid drachm of the solution of persulphate of iron an ounce of methylated spirit requiring 44 grains of the ferrocyanide of potassium dissolved in an ounce of water for complete precipitation. It frequently also contains nitric acid which may be detected by its odour, but more certainly on adding a little strong sulphuric acid and applying heat.

### LIQUOR HYDRARGYRI NITRATIS ACIDUS

The strength of this solution is sufficiently indicated by its density being about 2.0, and the freedom from mercurous salt by there being no precipitation of calomel on adding hydrochloric acid.

## LIQUOR LITHIÆ EFFERVESCENS

Lithia water should leave a residue of 5 grains from the evaporation of the contents of the usual half-pint bottle. It may contain lime from being made with hard water; the presence of soda or potash in place of the lithia or part of it would be detected by the 5 grains of residue being reduced to less than 4 by washing with f3iss of cold water; the residue should be freely soluble in HCl, and the solution should give no precipitate with oxalate of ammonium (lime) or with phosphoric acid and then ammonia in excess, which would yield a crystalline precipitate with magnesia.

It is perhaps too much to expect of lithia water that it shall contain no lime, as the B.P. does not direct it to be made of distilled water, and lime is naturally present in almost all water supplies; but if lime and magnesia are present in any notable quantity, the weight of the total residue ought in so much to be greater. The B.P. does not pointedly say that the lithia water shall not contain lime, but it requires that the 5 grains of residue shall answer to the tests for carbonate of lithium, and these tests include examination with oxalate of ammonium and with lime water.

## LIQUOR MAGNESII CARBONATIS

A little excess of carbonic acid is advantageous in tending to preserve this solution against loss of magnesia which is apt to be deposited as crystals at the bottom of the containing vessel. The test of strength given in the B.P. is most convenient when it is desired to test purity at the same time. A fluid ounce evaporated in water-bath should leave carbonate of magnesium weighing  $9\frac{1}{2}$  grains, reduced to 4 grains by ignition, and the latter should not yield any appreciable soluble matter to distilled water with which it may be shaken and from which it has been separated by filtration. Impurities are not commonly found in this preparation, though its strength is variable,

and if this point only is to be verified, a fluid ounce should yield a neutral solution with 11 grains of oxalic acid, but an acid one with 13 grains.

#### LIQUOR MORPHINÆ BIMECONATIS

The morphine which is thrown down from this solution by a small addition of solution of potash is freely soluble in excess of the caustic alkali but not of its carbonate. The red coloration with nitric acid is a morphine reaction; the acid should be used strong. The red colour produced with perchloride of iron is a reaction of meconic acid, and is so powerful as to cover the green tint which morphine gives with this reagent.

#### LIQUOR PLUMBI SUBACETATIS

Not subject to impurity. - Liable to loss of strength by absorption of carbonic acid, and precipitation of carbonate of lead. This change takes place to a very small extent with ordinary keeping. Its density is a satisfactory indication of its strength, and should be between 1.27 and 1.28. Precipitation may be used as an alternative;  $\mathfrak{z}$ ij in 2 ounces of water precipitated with 15 grains of oxalic acid dissolved in 2 ounces of water, and filtered, yields a further precipitate with 1 grain more of the acid, but, if again filtered, the reaction should be found complete.

#### LIQUOR POTASSÆ

This generally contains a little alumina, a little carbonate of potassium, and may contain traces of silica, lime, chlorides and sulphates, too little to be of any pharmaceutical importance. One fluid ounce should neutralise 30 grains of oxalic acid without effervescence; with an additional grain, the solution should redden litmus. The formation of a white precipitate during this operation would indicate lime. There would not exist in the same sample appreciable quantities of both lime and carbonate of potassium. The neutralising power and the approximate freedom from carbonate are the important points. See also Potassa Caustica.



## LIQUOR POTASSÆ EFFERVESCENS

Potash water of commerce is commonly made of less than the official strength, and some makers also add a little soda ; it generally contains lime from being made with hard water. The B.P. directs 5 ounces to be evaporated to 1 ounce and 12 grains tartaric acid added ; this should deposit 12 grains of acid tartrate of potassium. The total yield should be 14 grains, but about 2 grains will remain dissolved in the ounce of mother-liquor. On account of this solubility the precipitate should either not be washed, or should be washed only with a little dilute spirit (equal parts rectified spirit and water).

## LIQUOR SODÆ

This is liable to the same impurities as solution of potash and may be tested in the same way ; but, as regards strength, a fluid ounce should remain alkaline on the addition of 29 grains of oxalic acid, and become acid to litmus on the addition of another grain. The B.P. is more strict in its requirement of the absence of alumina in solution of soda than in solution of potash, and without any evident reason. The solution of soda being neutralised with nitric acid and evaporated to dryness, the residue should be soluble in water ; if silica were present it would remain undissolved in a gelatinous condition. If alumina should be present, which is not unlikely, ammonia would throw it down from the nitrate of sodium solution as a light translucent precipitate. The B.P. requires that solution of potash under this treatment shall be ‘unaffected or *but very slightly affected by ammonia.*’ But of solution of soda it says, ‘*not at all by ammonia.*’ For pharmaceutical use this is more strict than desirable.

## LIQUOR SODÆ CHLORINATÆ

The B.P. demands a much closer approach to the theoretical yield of available chlorine in this solution than in the solution

of chlorinated lime. A fluid drachm of the chlorinated soda solution with 20 grains of iodide of potassium, an ounce of water, and an ounce of hydrochloric acid forms a dark brown solution, the colour of which is reduced to a brownish yellow by the addition of 10 grains of hyposulphite of soda, and entirely decolorised by an additional half-grain. It would have been more reasonable to allow a little more latitude in this case, and a little less in the other. The presence of a little lime in the soda solution is not to be objected to, provided it is only what produces a slight precipitate on the addition of oxalic acid or oxalate of ammonium.

### LIQUOR SODÆ EFFERVESCENS

Soda water of B.P. strength should neutralise 1 grain of oxalic acid to each ounce, leaving the solution slightly alkaline to litmus after the carbonic acid has been driven off by heat or exposure to the air. Commercial soda water not intended for medicinal use may be expected to have from a fourth to a half of this strength.

### LIQUOR SODII ETHYLATIS

The most likely fault in this preparation is excessive causticity from the alcohol not being absolute, in which case the water it contains converts some of the sodium into caustic soda, and, if so, the gravity will be found much higher than 0.867, which is the official standard.

### LIQUOR ZINCI CHLORIDI

Not much subject to impurity. The common commercial qualities sold as disinfectants may contain traces of other metals, such as lead and iron, frequently present in the natural sources of zinc, and it should not be assumed that earthly and alkaline chlorides will be absent, or that the preparation made for disinfecting purposes is fit for pharmaceutical use. Pharmaceutical solution of chloride of zinc should, when acidu-

lated with HCl, produce no precipitate with  $\text{H}_2\text{S}$ , otherwise copper, lead, or both are probably present, and when the solution containing  $\text{H}_2\text{S}$  is rendered alkaline with ammonia, the precipitate thrown down should be white, otherwise iron is probably present ; and if the  $\text{H}_2\text{S}$  or sulphhydrate of ammonium be added so long as it throws down a further precipitate and the whole thrown upon a filter, the filtrate should contain only ammoniacal salts, and on evaporation the residue should entirely volatilise below red heat, otherwise earthy or alkaline salts are present. The strength of the solution is sufficiently indicated by its sp. gr. being 1.46.

### LITHII CARBONAS

The statement in the pharmacopœia that lithium carbonate is soluble in 150 parts of cold water, while literally correct, is calculated to mislead. In three determinations 100 grains of solution of carbonate of lithium saturated at  $60^\circ \text{F}$ . yielded me 1.3—1.25, and 1.3 of carbonate dried at  $212^\circ \text{F}$ . The average is thus 1.28 of carbonate in 98.72 of water, or 1 of carbonate in 77.1, or little more than half the quantity of water which the B.P. statement might lead us to think necessary. With this correction the characters and tests of the B.P. give a fair indication of the purity of the salt. The above saturated solution on being boiled deposited a crystalline precipitate of the carbonate of lithium ; 90 grains of the boiled solution on evaporation yielded 0.95 carbonate to 89.05 of water, or 1 part soluble in 93.7 of boiling water. A ready method of obtaining general evidences of purity from this solubility is to shake 3 or 4 grains in an ounce of water ; it should entirely though slowly dissolve, giving evidence that earthy impurities and salts of the heavy metals are absent. If half a drachm of the carbonate is shaken with two drachms of distilled water and filtered, 85 minims of the filtrate on evaporation should leave not more than 1 grain of residue, showing the absence of all readily soluble carbonates. The change of weight occasioned by conversion into sulphate is greater than would occur with any other carbonate ; so, if the product amounts to the pro-

portion the B.P. indicates, it is presumptive evidence of its identity and proximate purity.

Carbonate of lithium		by conversion into dry sulphate gains	
		about 50% in weight	
„	magnesium	„	25% „
„	calcium	„	36% „
„	sodium	„	34% „
„	potassium	„	26% „

The B.P. testing of a solution in sulphuric acid with oxalate of ammonium and solution of lime, which would indicate respectively lime and magnesia, is unnecessary if the three or four grains dissolve entirely in an ounce of water.

### LITHII CITRAS

The B.P. tests for citrate of lithium, like those for the carbonate, depend upon the low equivalent of this metal. The complete combustion of no other citrate would result in so small a residue of carbonate as is the case with lithium; consequently the residue of only 7·8 of white carbonate of lithium from 20 grains of the citrate is presumptive evidence that the salt yielding this result is citrate of lithium. But it must be remembered that a mixture of citrate of potassium or sodium with citrate of ammonium might give a similar result as regards weight, or a contamination of all these with citrate of lithium might do the same, and not be detected unless the residual carbonate were examined as described under lithium carbonate. The burning of the citrate is not very readily performed. The injunction to burn at a low red heat is probably intended to avoid the fusion of the carbonate, which takes place at a full red heat, and when the unburned carbon becomes mixed with the fused carbonate it protects it from oxidation. If the combustion be conducted at a low red heat, it is very slow unless completion be aided by throwing in small portions of yellow oxide of mercury, which readily supply the required oxygen without leaving any fixed residue. A porcelain capsule is to be preferred for this operation, as lithia is not without action upon platinum.



## MAGNESIA

The B.P. provides that calcined magnesia shall be free from lime and sulphuric acid, impurities rarely present in more than minute traces. The common impurities of almost all commercial samples are black specks—no doubt furnace dust acquired in the process of calcination, and scarcely visible till on mixing with water and decanting the light portion, the mechanical impurities are found at the bottom of the vessel. The frequency and the facility with which furnaces are now heated by means of gaseous fuel should afford us the means of having magnesia free from this impurity.

## MAGNESII CARBONAS

The carbonates of magnesium of commerce, both heavy and light, are salts of great purity. The B.P. tests are directed against lime, sulphuric acid, and traces of the heavy metals such as iron, lead, or zinc, which would be precipitated by  $H_2S$  in an alkaline solution. They may be looked for but are rarely found.

## MAGNESII SULPHAS

This is usually a very pure salt. Occasionally it may be found to contain traces, or more than traces, of protosulphate of iron, the presence of which is best detected by the addition of a little chlorinated solution of soda, which oxidises the iron and precipitates it as ferric hydrate. The U.S.P. adopts sulphhydrate of ammonium and ferrocyanide of potassium as the tests for metals; they cover a greater number of impurities, and are even more delicate than the chlorinated solution of soda, but any one of the three may be considered satisfactory. The simple precipitation with a caustic alkali without an oxidising action might fail to give satisfactory evidence of iron if present in small quantity. The reactions with chloride of barium and with chloride of ammonium and phosphate of soda

are given simply as the reactions of a sulphate and of a magnesium salt. The precipitation by carbonate of sodium would indicate the probable presence of sulphate of sodium if the yield of calcined magnesia were less than 16 per cent., but the boiling of the solution after the addition of the carbonate of sodium is essential, otherwise some magnesia is held in solution by the carbonic acid. It must also be remembered that the quantity of magnesia may vary a little from the theoretical yield, as a consequence of the sulphate containing a little more or less than its legitimate quantity of water.

### MANGANESII OXIDUM NIGRUM

Native black oxide of manganese usually contains a considerable contamination of iron, which is not important when it is used only for the evolution of chlorine ; but for internal use a precipitated oxide should be preferred.

### MEL

The B.P. provides against honey being mixed with starch. By boiling the honey with water the starch becomes dissolved and capable, when cold, of producing the well-known blue-black precipitate with a drop of tincture of iodine. Starch is not a common impurity—glucose and cane sugar are more to be anticipated, but are not so readily detected. In the U.S.P. the solubility of honey in spirit is used to indicate its freedom from glucose thus :—A fluid drachm of honey is mixed with an equal bulk of water and added gradually to five fluid drachms of absolute alcohol ; the solution should be not more than faintly opalescent.

### MENTHOL

Commercial menthol in crystals is usually contaminated only with a little of the fluid oil from which it has been deposited, but that supplied in cones is frequently hardened with wax or paraffin, which may be detected by the menthol gradually

evaporating at a water-bath temperature, and if thus adulterated a fusible residue remains nearly devoid of odour.

### MORPHINÆ ACETAS

Acetate of morphine has an acetous odour from the gradual escape of acetic acid, which it slowly loses by keeping. The B.P. provides against any considerable loss of acetic acid by requiring that 20 grains in 3j water shall form a slightly turbid solution rendered clear by one grain of acetic acid. It is to be regretted that an ambiguous term should be used, as it leaves room for doubt as to whether real acetic acid is intended, or the 33 per cent. solution which is officially known as acetic acid in the Pharmacopœia.

The custom in the B.P. is to use the term 'grains by weight' in speaking of the official forms of acetic acid, and in many other cases when speaking of liquids the term 'grain measures' is used, but only in one instance is the term 'grain' used without qualification in speaking of acetic acid, and that is in defining the quantity of *real* acetic acid contained in a fluid ounce of dilute acetic acid. We might therefore conclude that a grain of acetic acid in this instance means a grain of the absolute acid, though this is more than should be required with 20 grains of any ordinary sample of acetate of morphine.

That the solution 'should be rendered clear by a drop of the 33 per cent. acid' would be a better expression of the requirement. It is also provided that the solution when precipitated by a slight excess of ammonia shall yield a precipitate which, when washed and dried in a water-bath, shall weigh 15 grains. Less than this quantity might result from excess of moisture or some other impurity in the acetate, and more than this yield might imply loss of water as well as loss of acetic acid, in the drying or keeping of the salt. Even skilful operators may fail to get results as exact as the wording of the pharmacopœia implies.

A good additional check upon the purity of morphine and its salts is found in the free solubility of morphine in lime-water. One grain of the acetate dissolved in half a drachm of water

should give a copious precipitate with ʒj lime water, and should form a clear solution on increasing the lime water to ʒij or ʒiij. This test provides against the presence of narcotine and a considerable number of other alkaloids which, though not probable, are possible contaminations.

The B.P. test by weighing would have been more complete had it included the further requirement that, after drying and weighing on the filter, a further washing with fʒss of ether or benzine should not cause any appreciable loss of weight. Where there is sufficient ground for estimating the amount of morphine precipitable from any sample of its salts, the further washing of the precipitate with ether or benzine should not be omitted.

### MORPHINÆ HYDROCHLORAS

The comments on testing acetate of morphine sufficiently cover the requirements of the hydrochlorate, except to note that the latter is required to yield 16 grains of precipitated morphine, from ʒj instead of 15 grains.

### MORPHINÆ SULPHAS

The precipitate yielded by ammonia from ʒj of sulphate of morphine is practically the same as that from the hydrochlorate; but in the examination with lime water the sparing solubility of calcium sulphate must be borne in mind, and any error from this circumstance may be guarded against by adding an equal bulk of distilled water to the lime water and, if necessary, filtering to separate any carbonate thrown down.

### OLEATUM HYDRARGYRI

The chief defect this preparation is subject to, is the reduction of the oxide of mercury from the abstraction of oxygen by the oleic acid. Metallic mercury, in the form of a dark precipitate, results from long keeping, and is speedily produced by the action of heat. The B.P. requires that a gentle warmth



should not produce this action. It is most readily produced if the oleate is made from impure oleic acid.

### OLEUM OLIVÆ

The pharmacopœial characters of olive oil do not include anything calculated to detect its adulteration with other vegetable oils. An old and well-known test is to submit the sample to the action of nitric acid, or to make a small sample of nitrate of mercury ointment with it, most of the inferior seed oils making a dark-coloured soft or semi-fluid compound, while the pure olive oil yields a pretty firm ointment of pale orange, inclining to lemon colour. When wanted simply as a test for the oil, the operation may be conveniently performed thus :

Into a half-pint flask or dish, pour an ounce of the oil, and heat it to about  $212^{\circ}$  F., then pour in fʒj of B.P. nitric acid, (1.42 gravity) and agitate till brisk effervescence is set up, maintain the heat till the action is completed, as indicated by the cessation of the effervescence, and then allow it to cool. The product should be firm and bright-coloured. The colour is perhaps best described as a pale orange, inclining to lemon, while a darker orange, inclining to brown, is commonly produced by samples adulterated with rape, cotton seed, or some other vegetable oils. The texture or mechanical condition of the product is also characteristic, that from the pure olive oil being of a rather spongy character, breaking short when dug up with a knife, while the inferior oils yield a semi-fluid or pasty product, more like the consistence of soft soap.

The nitrate of silver test, as described under "Adeps," is also applicable to olive oil, and yields the brown coloration of the cotton seed reaction with a smaller percentage of the impurity than would be detected by the above test.

### OPIUM

The analysis of opium has been the subject of so many critical papers, that it would be impracticable to give any profitable sketch of the methods advocated, or any comparison

of their merits in a short notice. The B.P. method acts fairly well, and gives pretty constant results, as far as I have tried it, but that particular process of analysis is not insisted upon, permission being given to use *any trustworthy method*. The limitation of the strength between 9·5 and 10·5 per cent. is a reasonable and practical one, and we might say this more emphatically if it was defined as being the percentage of morphine, capable of being obtained by that particular process. There are certain losses involved in every process ; what they amount to in this case, and in sundry others, is a debateable point, and if it were found that some other method would yield 9·5 per cent. of morphine with a particular sample of opium which by the B.P. process only yielded 8·5 or 9·0, that sample would be sanctioned by the present rule, though if the rule were that the yield must come between 10·5 and 9·5 by the process described, the weak sample *containing*, but not *yielding* 9·5 would be rejected.

### PARAFFINUM DURUM

The sp. gr. of paraffin may be tested as described under wax : 33 grains should float with a 2 grain weight attached, but sink with 3 grains. And the melting point may also be taken as there described, and may vary between 110° F. and 145° F. Paraffin, so long as it is free from odour, can scarcely be said to be subject to impurity, the gravity and melting points being variable from the difference in the proportions of the constituents always present.

### PARAFFINUM MOLLE

The sp. gr. of soft paraffin cannot be estimated as described under wax, but is sufficiently and conveniently indicated by weighing an avoirdupois ounce into a graduated measure and heating it to the melting point ; it should then measure between f̄3ix and f̄3ixss.

The melting point is a more important matter. The B.P. requires it to be between 95° F. and 105° or a little more ; but

commercial specimens, with melting points such as these, are sometimes made by mixing the very soft or fluid paraffins with some of higher melting point with the disadvantage that the fluid part separates.

Paraffinum Molle may be tested for this fault by digging a hole a few inches deep in its surface, in which no oil should accumulate in the course of a day at ordinary temperatures. From the low price at which paraffins can now be obtained, the adulteration with saponifiable fats is a contingency against which it is scarcely necessary to make provision.

### PEPSIN

The official directions for estimating the activity of pepsin may be considered a condensation of Mr. Benger's instructions, and we may benefit by his fuller directions. At its best the process has several points of uncertainty. Mr. Benger recommends the white of egg, after being pressed through the sieve, to be rubbed in a glass mortar with the acidulated water to insure its not being clotted together, and this is no doubt an advantage, but the degree of rubbing as well as the degree of stirring or shaking which it is subject to while the digestion is going on, influences the result, and the degree of clearness which is looked for before it is considered that solution has been effected, is a point not capable of definition ; little particles of insoluble matter will be found floating in the fluid for a long time after the great bulk of the albumen has been dissolved, and if these are very small it would be an injustice to the sample to condemn it on account of solution not being perfect at the expiration of the prescribed half-hour. Nothing but repeated experiments can give the operator a satisfactory power of judging the results. I prefer to have the hard-boiled white of egg rubbed through the sieve, and then rubbed in a mortar with the acid and water, transferred to a bottle, corked, shaken, and heated in a water-bath to 130° F., and then add the pepsin diffused through a little water, repeating the shaking every five minutes and immersion in the bath

during the intervals till the expiration of the half-hour, and if solution is imperfect at the end of the time to strain the liquor through a small filter of doubled muslin, washing a little water through it, and then drying and weighing the residue, using the under piece of muslin as a counterpoise, the difference in their weights giving the weight of undissolved albumen. This weight will frequently be found quite insignificant, even when there is a palpable amount of imperfectly dissolved albumen ; the weight of dry albumen, multiplied by 7, gives approximately the weight of white of egg left undissolved.

### PHYSOSTIGMINA—PILOCARPINÆ NITRAS

The alkaloids of calabar and jaborandi are not yet so thoroughly known as to afford us ready means of detecting impurities they may contain ; consequently the B.P. characters, which are intended for their identification, make no pretence of proving their purity, but the more closely their reactions correspond with the official requirements, the less probability there is of any considerable fault.

### PILULA HYDRARGYRI

As most pharmacists will buy their blue pill rather than make it, a few tests of its quality become more important than if it were customarily a home-made pill-mass. The faults to be anticipated are deficiency of mercury or any considerable oxidation of the same, though it is much less liable to oxidation than grey powder. If it contain its legitimate proportion of mercury in the metallic state, 30 grains rubbed to a soft pulp with a little water, a scruple of iodide of potassium, and 12 grains of iodine should after a little trituration lose its brown iodine colour, the iodine and mercury having combined to form the periodide, and this with the iodide of potassium forms the pale yellow double salt. The colour reaction is rather obscured by the conserve of roses and licorice powder contained in the mass, but not so much so as to leave any considerable



portion of free iodine unnoticed, and even a small trace of iodine may be detected if the pulp be further diluted with water and filtered. A strong brown colour disappearing on the addition of a grain or two of hyposulphite of sodium would indicate iodine, and consequently less than the legitimate proportion of metallic mercury. The theoretical quantity of iodine which should be taken up by this reaction is 12·7 grains, but a result very close to theory is not to be looked for in a body of this nature.

### PLUMBI ACETAS

The recrystallised acetate of lead of pharmacy is usually a very pure salt. By keeping, it loses a little water and acetic acid, and becomes slightly basic or carbonated, and imperfectly soluble in pure water or water holding carbonic acid in solution.

### PLUMBI CARBONAS

What may be called the natural impurities of lead, i.e. silver, antimony, iron, copper, and tin, do not occur in the commercial carbonate in more than traces too small to interfere with its medicinal uses. Commercial white lead, though generally a satisfactorily pure salt, is occasionally found adulterated with sulphate or carbonate of barium or calcium, or sulphate of lead; a general indication of purity may be obtained by the following tests. Half an ounce of distilled water shaken with ʒj, the sample filtered and the filtrate evaporated, should not leave any notable residue; the sample treated with acetic acid should totally dissolve, showing the absence of the sulphates; if the solution in acetic acid is diluted with two or three volumes of water, and sulphuretted hydrogen passed through it till no further precipitate is thrown down (or till a portion filtered off has an odour of the gas), the filtrate should evaporate without any notable residue; this will show the absence of earthy carbonates, &c.

## PLUMBI IODIDUM

Iodide of lead, if precipitated from acetate, may contain oxide of lead ; if precipitated from nitrate containing free acid, it may contain free iodine. The latter impurity may be detected by its odour and by the darker colour of the iodide. The excess may be dissolved out by treatment with spirit. The former impurity, if in any conspicuous quantity, gives the iodide a paler appearance, and may be dissolved out by treatment with acetic acid. If the iodide be pure, the acetic liquor after filtration should be colourless (free from iodine) and give a very small precipitate on the addition of sulphuric acid (free from oxide). Two grains of the iodide should entirely dissolve in 1 ounce of boiling distilled water, and on cooling  $\frac{9}{10}$  of this should separate as golden scaly crystals.

## PLUMBI OXIDUM

Litharge may be expected to contain all the impurities which occur in commercial lead ; and these are numerous, though in quantities too small to have any pharmaceutical importance. Commercial lead of ordinary quality contains from 99.92 to 99.98 per cent. of pure lead, the impurities consisting of copper and antimony with traces of iron, zinc, and silver. But as lead of high purity does not so readily oxidise in the furnace, it is customary to add a little inferior lead or 'slag lead,' containing 3 or 4 per cent. of antimony to the furnace charge, which much facilitates the production of the litharge, but, of course, detracts a little from the very high purity which it might otherwise possess. Litharge should be entirely soluble in dilute nitric acid without effervescence (carbonates), and the solution should be free from colour (proximate absence of copper and iron). The solution being treated with *excess* of sulphhydrate of ammonium throws down a copious black precipitate of sulphide of lead, the filtrate from which, when acidulated with HCl, throws down a precipitate of sulphur

almost white ; an orange tint would indicate antimony. The filtrate from this precipitate, when evaporated to dryness, should leave a residue entirely volatilised below red heat, showing the absence of earths and alkalies. From the customary purity of litharge this examination is uncalled for.

### POTASSA CAUSTICA

Under tests a verbal error occurs in B.P. It is scarcely necessary to caution the student not to add nitrate of silver *and* chloride of barium to the acidified solution, but for 'and' read 'or.' Fifty-six grains of KHO should neutralise 63 grains of oxalic acid or 1000 grain measures of volumetric solution of oxalic acid. The B.P. requires that 56 grains shall require at least 900 grain measures of the volumetric acid, thus allowing 10 per cent. as the limit of the impurities it may contain. These impurities should consist almost entirely of water, carbonic acid, and a little alumina or silica, and these impurities are not of much importance except in the negative sense of subtracting so much value ; but commercial samples from the best sources often fall short even of the B.P. neutralising power. The theoretical percentage of water is 16 per cent. (old form KO, HO), but Davy found by experiment 17 to 18 per cent., Gay Lussac 18.45 to 20.72, Darcet 27 to 28 per cent., and Remington says the commercial article rarely contains less than 30 per cent. of water. This latter figure gives it 86 per cent. of its theoretical neutralising power. Nitrate and nitrite of potassium are also impurities sometimes found, which the official tests do not provide against. Dunstan found 1 per cent. of nitrate in commercial caustic potash, and found on an average from 78 to 79 per cent. of 'total alkali' (presumably KHO), while chloride, alumina, and silica amounted to 4.5 per cent.

To examine for nitrate, take 5 grains of the sample, dissolve in f3i of water, add a small fraction of a grain of KI and enough dilute sulphuric acid to give an acid reaction. If nitrate be present the yellow or brown colour of iodine will be immediately developed.

It must be noted that the B.P., under the heading 'Prepara-

tion containing caustic potash,' gives 'liquor potassæ 27 grains in a fluid ounce.' But under 'Liquor Potassæ' the strength required is that the fluid ounce shall contain 27 grains of *absolute* KHO, not 27 grains of 'caustic potash B.P.,' nor of commercial caustic potash.

## POTASSA SULPHURATA

This is a somewhat indefinite chemical, and variable both in appearance and composition according to its age and keeping. It is liver-coloured when new and greenish after keeping, and the change from the former to the latter colour proceeds from the surface to the interior. The green portion being only skin-deep implies a satisfactory condition, but a considerable penetration of the green colour does not imply that the sample is worthless, for the green portion itself contains a large percentage of soluble sulphide. The B.P. test of about 50 per cent. being soluble in rectified spirit is enough, and probably more than most pharmacists will consider is demanded in the way of quantitative examination. The U.S.P. requires that 10 parts of sulphurated potash triturated with 12.69 of crystallised sulphate of copper and 60 of water and filtered should give a filtrate which contains no copper, as indicated by no further black precipitate being produced on the addition of sulphhydrate of ammonium, thus indicating the presence of 56 per cent. of true sulphide of potassium. A more satisfactory indication would be that the filtrate should continue to contain soluble sulphide, as shown by its yielding a further *black* precipitate with more sulphate of copper. The sulphurated potash contains some carbonate, which will precipitate carbonate of copper, this being in turn converted into sulphide if there be sufficient sulphide of potassium present; but, if not, part of the copper is filtered out as carbonate, and its absence in the filtrate does not indicate that the whole of the copper has been converted into sulphide. But the appearance of soluble sulphide in the filtrate implies that the sample is so rich in sulphide as to precipitate all the copper as sulphide, and leave a little to spare.



I would suggest as a fair test of quality that 10 grains of the sulphurated potash triturated with 10 grains of crystallised sulphate of copper, and a drachm of water, forms a black magma, a drop of which thrown on blotting paper produces a black spot surrounded by a nearly colourless margin, the margin becoming blackened if touched with solution of lead. It is convenient to let a drop of liquor plumbi fall upon the paper so near to the test that it spreads into the pale margin, and shows the colour where the two drops meet.

### POTASSII ACETAS

Not much subject to impurity except water, as a result of its deliquescence. The deep red colour which it produces with perchloride of iron is indicative of the formation of ferric acetate by double decomposition. Its neutrality to test paper and almost perfect solubility in rectified spirit exclude carbonate of potassium, a possible impurity, and the absence of change with sulphhydrate of ammonium shows that it is not contaminated with iron, lead, or copper.

### POTASSII BICARBONAS

This is usually a very pure salt, a little hygroscopic moisture or a little deficiency of carbonic acid being the most probable fault. Fifty grains in half a pint of boiling water with 31 grains of oxalic acid should be faintly alkaline, and become acid with an additional grain of oxalic acid. A deficiency of carbonic acid increases the hygroscopic character of this salt, and as a consequence the less carbonic acid the more water, and the neutralising power may remain the same; and if the loss of weight by ignition be found to be 31 per cent. both tests fail to indicate the fault. The U.S.P. tests for deficiency of carbonic acid by mixing solutions of chloride of barium and bicarbonate of potassium in water sufficient to hold in solution the barium bicarbonate which should result. If there be deficient carbonic acid present, a portion of barium mono-

carbonate is formed, which is immediately precipitated. The test may be conveniently conducted thus : dissolve 10 grains of the bicarbonate of potassium in 5 ounces of cold water, and  $10\frac{1}{2}$  grains of barium chloride in 5 ounces of cold water ; mix the solutions without violent agitation ; no precipitate or opalescence should make its appearance within ten minutes.

### POTASSII BICHROMAS

This salt, when recrystallised for pharmaceutical use, is usually very pure, not calling for any special examination.

### POTASSII BROMIDUM

Like most other potassium salts the bromide precipitates cream of tartar on the addition of tartaric acid, though complete decomposition is not thus effected. When its solution is mixed with chlorine (or bleaching powder and hydrochloric acid) bromine is liberated. If chloroform be now added, it falls to the bottom carrying with it the bulk of the liberated bromine. Twelve grains of the bromide, with a fraction of a grain of yellow chromate of potassium in two ounces of water, should produce a white precipitate of bromide of silver with  $16\frac{1}{2}$  grains of nitrate of silver, and an additional grain throws down the red brown chromate of silver. The appearance of the red colour with the former quantity of silver would suggest the presence of iodide, which from its higher combining proportion would sooner be saturated, and leave some silver to combine with the chromic acid, and a failure to produce the red colour with the additional grain would suggest chloride, which from its lower combining proportion would continue to precipitate silver as chloride or mixed chloride and bromide for some time longer before any would be left free to combine with the chromic acid.

Besides iodide and chloride, bromate, sulphate, and carbonate are impurities which may be anticipated. The official test for iodine is to liberate it with bromine or chlorine and

add starch paste, which gives blue colour if iodine be present, but yellow if the bromide be pure. Pharmaceutically it is generally more convenient to acidulate the bromide solution with HCl, and oxidise with a drop of permanganate, or a small addition of chlorinated lime or chlorate of potassium, which will develop the iodine brown colour if this element be present, and if any confirmation be desired a drop or two of chloroform shaken with it speedily sinks to the bottom coloured violet with the iodine. In testing for carbonate and sulphate the B.P. directs the use of *saccharated* solution of lime, and *nitrate* of barium, without any evident reason for deviating from the use of the more commonly adopted reagents—lime water and chloride of barium. The absence of bromate is indicated by there being no immediate production of yellow colour (liberation of bromine) when the solution of bromide is acidulated with sulphuric acid.

In the B.P. the presence of chloride is only provided against by the application of the volumetric test, but this would fail to give the indication if moisture or other impurity not capable of precipitating silver were there in such quantity as to counterbalance the greater precipitating power of the chloride. A better test for chloride is to take 12 grains of the bromide in 3j of water; add a solution of ammonio-nitrate of silver in excess (say 18 grains of nitrate of silver dissolved in  $\frac{1}{2}$  ounce of water and liquor of ammonia sufficient to redissolve the precipitate at first formed); on mixing these solutions a copious precipitation of bromide of silver will take place while any chloride present will remain in solution together with a trace only of bromide, the whole being thrown upon a filter and the precipitate washed with water enough to make up an ounce of filtrate; add to this filtrate nitric acid sufficient to give it an acid reaction, and any chlorine present will be thrown down as chloride of silver which after washing and drying may be weighed. As bromide of silver is not totally insoluble in weak ammoniacal liquors, there is always a precipitation of bromide of silver on adding the nitric acid, but operating as above it does not weigh more than 0.05. It must also be noted that strongly ammoniacal



liquors have a stronger solvent power ; an ounce of '959 ammonia dissolving one grain of the bromide ; but with ordinary care operating as above the final precipitate will not amount to 0.1 unless chloride be present.

An experiment, as a check upon this, was performed with twelve grains of bromide of potassium to which was added 0.1 grain of chloride of sodium ; the final precipitate thrown down by nitric acid in the ammoniacal solution was in this case 0.3, agreeing fairly well with a yield of 0.24 of chloride of silver theoretically present, and 0.05 of the bromide.

### POTASSII CARBONAS

The common impurities of carbonate of potassium are silica, alumina, chloride and sulphate of potassium, and from its hygroscopic quality a greater amount of water than the B.P. allows. Carbonate of potassium crystallises with two equivalents of water = 20.7 per cent., but it is more convenient to carry the evaporation so far as to produce a sandy condition of rough powder on cooling, and this condition agrees with 16 per cent. or less of water ; the percentage in any sample may be determined by heating 100 grains to redness in a platinum capsule, keeping the same carefully covered while it cools and weighing without loss of time or exposure to the air. It is however sufficient for all ordinary requirements to know that the carbonate should be in a loose sandy condition. The saline and earthy impurities are usually too small to be of any importance in its pharmaceutical use.

No.

### POTASSII CHLORAS

The commercial salt is generally almost pure, only traces of chloride of potassium or calcium being indicated on testing with nitrate of silver or oxalate of ammonium. The B.P. does not sanction any trace of either. The U.S.P. permits a trace of chloride which is perhaps reasonable, but requires the total absence of lime.



## POTASSII CITRAS

Pharmaceutical citrate of potassium, which is white and perfectly soluble, rarely contains any impurity worth noting, though it may contain a trace of lead from the citric acid, in which case its solution will become darkened on the addition of a drop of solution of sulphhydrate of ammonium. If the citrate does not form a bright solution with its own weight of water, earthy impurities may be suspected to have been introduced through the use of an impure carbonate of potassium in its manufacture. If it be desired to apply the equivalent of the B.P. volumetric test, 26 grains may be heated to full redness in a small capsule ; when cold, 16 grains of oxalic acid and 2 drachms of water should give a neutral solution so soon as the effervescence has gone off. The pharmacopœial instruction to wash the charred mass and filter the solution before estimating the alkali it contains, has the theoretical advantage of titrating only the alkali and leaving in the charcoal any carbonate of calcium, which may be present in small quantity from the original salt containing calcium citrate as an impurity ; but it has the practical disadvantage of involving considerable chances of loss of alkali in the charcoal and the filter, probably amounting to as much as the traces of calcium salt likely to be found in the citrate. It is better to operate as above, and test the citrate for lime, with ammonium oxalate. The presence of the charcoal does not make the litmus colour difficult to observe. An acid reaction with these proportions of reagents is to be anticipated, as the B.P. does not make allowance for hygroscopic moisture which it is scarcely practicable to exclude.

## POTASSII CYANIDUM

The B.P. description of cyanide of potassium is not by any means satisfactory.

The process by which it is prepared does not yield a pure product, a circumstance which is of little moment if the cyanide is to be used only for the purification of bismuth, the only use

which the B.P. indicates ; but as it is also occasionally used medicinally, the instructions should have directed that when required for medical purposes it should be purified by solution in, and crystallisation from spirit.

The odour of HCy which it is described as possessing, is only the consequence of its absorbing carbonic acid and water from the air, and liberating hydrocyanic acid. The requirement that it shall be almost entirely soluble in absolute alcohol, indicates the absence of carbonate of potassium, a common impurity ; but as the cyanide is itself only sparingly soluble, the alcohol must be used in large quantity, about 250 parts of alcohol to one of cyanide being necessary for solution. The equivalent of the volumetric examination may be performed by dissolving 10 grains of the cyanide in an ounce of distilled water, adding  $12\frac{1}{2}$  grains of nitrate of silver, which should form a clear solution, and an additional half-grain of the nitrate should produce a permanent precipitate. With the commercial fused cyanide, the precipitate usually becomes permanent with the first 12 grains of the nitrate. The U.S.P. allows 10 per cent. of impurity, which is more in accordance with the results obtainable with the fused cyanide.

### POTASSII FERROCYANIDUM

The B.P. gives the prominent characters of this salt, but not tests for impurities ; commercially it is usually obtained so nearly pure as to require no examination at the hands of the pharmacist.

### POTASSII IODIDUM

The impurities to be anticipated are chloride, bromide, and iodate of potassium. The B.P. sanctions a trace of free alkali ; without this the salt is more prone to become yellow by keeping, probably as a result of atmospheric ozone liberating iodine. If tartaric acid be added to a solution of pure potassium iodide, hydriodic acid is liberated, but if iodate be also present in the sample under examination, iodic acid is also liberated at the

same moment, and, reacting on the hydriodic acid, iodine is set free. This is an important reaction, as a change of this kind would take place in the stomach with such a sample, and the patient would be under the action of free iodine, a much more potent agent than that intended. Ten grains of the iodide in an ounce of water on the addition of 10 grains of nitrate of silver, leaves a little of the iodide still in solution, and capable of yielding a further precipitate on the addition of  $\frac{1}{2}$  grain more of the nitrate, but on the subsidence of this second precipitate, the liquor does not again become clouded with more of the reagent, unless chloride or bromide be present as an impurity. These latter salts having lower equivalent weights, any admixture of them will increase the quantity of nitrate of silver which will be precipitated by 10 grains. This cannot be regarded as a delicate test for these impurities, but for medical purposes it is practically satisfactory, as they are therapeutically only diluents of the more active salt. A better test for Br or Cl is founded upon the solubility of their silver salts in liquor of ammonia, and the comparative insolubility of the iodide of silver in the same liquor. Dissolve 10 grains of the iodide of potassium in  $\frac{1}{2}$  ounce of solution of ammonia, add 11 grains of nitrate of silver previously dissolved in a little water, mix and filter ; to the filtrate, which contains silver chloride or bromide if such be present, but a mere trace of iodide, add nitric acid in slight excess ; any chloride or bromide which had been dissolved in the excess of ammonia will now be precipitated, promptly if in considerable proportion, slowly if less than 1 per cent.

### POTASSII NITRAS

Refined nitre of commerce generally contains both chloride and sulphate, and the re-crystallised salt known as 'needles' is rarely quite free from them, though the quantity is too small to be of any importance for medical use. A slight haziness of the solution when treated with nitrate of silver or chloride of barium, need not condemn the sample for any purpose except chemical analysis.

## POTASSII PERMANGANAS

The crystallised salt of commerce is usually nearly pure, and cannot be said to demand any examination at the hands of the pharmacist. The B.P. directs that 5 grains of the permanganate shall require 44 grains of granulated sulphate of iron for complete decoloration aided by fʒij of dilute sulphuric acid. Commercial granulated sulphate is not reliable enough to permit of this test having much value without a previous assay of the iron salt. A well-crystallised permanganate is about as reliable as any of the salts with which it can be assayed.

## POTASSII SULPHAS

The official tests for this salt are intended rather for its identification than for the detection of impurities ; the salt as found in pharmacy does not require examination.

## POTASSII TARTRAS

Not much subject to impurity. It may contain rather more or less than its normal water, or potash ; if correct in these particulars 61 grains heated to redness in a platinum capsule should form a neutral liquor with 31 grains of oxalic acid. The washing of the charred mass previous to treating it with acid is not essential, and risks some loss ; a better method is so soon as the capsule is cool enough to handle, to put it into a beaker with water enough to cover it, add the oxalic acid, and heat the liquor so long as effervescence continues, after which it may be tested with litmus paper without filtration.

## POTASSII TARTRAS ACIDA

Commercial cream of tartar generally contains a small percentage of calcium as tartrate, and occasionally also salts of barium. Twenty grains in an ounce of distilled water should entirely dissolve with the aid of heat, and should not yield precipitate on the addition of a few drops of sulphuric



acid. The B.P. sanctions the presence of a little lime, which is a natural impurity not easily removed, and not of serious moment, but barium should be rigidly excluded, and would be left undissolved in the treatment with hot water if present as sulphate, or would be precipitated on the addition of sulphuric acid if present as any soluble salt.<sup>1</sup> The volumetric examination is not of great value ; if lime be present in unusual proportion, the deficiency of carbonate of potassium in the charred residue would be indicated by less than the normal quantity of oxalic acid being required to neutralise it. If tartrate of sodium be present, the lower equivalent of sodium will require a larger proportion of oxalic acid for its neutralisation, and if calcium, sodium, and potassium be all present, the sample, though unusually impure, might answer the volumetric test. The neutralisation test may be performed as follows: 51 grains of cream of tartar heated to redness in a platinum crucible till it ceases to flame, leaves a residue which when well washed with water yields an alkaline solution which continues faintly alkaline on the addition of 15 grains of oxalic acid, but is rendered acid to litmus on adding two grains more of the acid. In this case it is necessary to wash the charcoal and filter the solution, as carbonate of lime, which it is almost certain to contain, should be estimated separately if at all. The calcium tartrate is converted by burning into carbonate, and the cinder after washing to remove the potassium carbonate will still produce effervescence if treated with 2 grains of oxalic acid, and the resulting liquor will have an acid reaction, unless the proportion of calcium tartrate in the cream of tartar is more than should be allowed.

Twenty grains of bicarbonate of sodium with 47 grains of pure cream of tartar should make a clear solution faintly acid.

<sup>1</sup> As alkaline tartrates retard the precipitation of barium by sulphuric acid, the application of heat and time should be allowed. In a more critical examination of cream of tartar, strongly igniting the sample would convert any barium compound it might contain into barium carbonate. After washing the charred mass with water, hydrochloric acid would extract the barium, when it might be detected or estimated in the usual way.

## QUININÆ HYDROCHLORAS—QUININÆ SULPHAS

The quinine salts of commerce usually contain more or less of the other cinchona alkaloids which are not entirely separated in the process of manufacture without considerable difficulty. The B.P. allows 'not much more than 5 per cent. of the sulphates of these alkaloids,' and gives somewhat elaborate processes for their estimation, which it is unnecessary to repeat. In the 'general characters' it is stated that quinine should dissolve in pure strong sulphuric acid with only a feeble yellow colour not increased by gentle warming. This is intended to guard against the presence of many organic matters which would blacken by this treatment. The requirement that the sulphate lose 15.2 per cent. of its weight by drying at 212° F. is intended to show that it has not lost water of crystallisation, which it is apt to do by keeping. The complete combustion of the salt without leaving ash is intended to indicate its freedom from inorganic impurities such as lime or fixed alkalies. The U.S.P. requires that sulphate of quinine shall lose not *more* than 16.2 per cent. of water by drying at 212° F.

Though much has been written on the testing of quinine, a good and ready mode of assay is yet a desideratum. If 10 grains of sulphate of quinine, 10 grains of precipitated carbonate of barium, and half an ounce of water be macerated for half an hour and filtered, 100 grains of the filtrate should leave on evaporation less than 0.1 grain of residue. And if the filtrate be previously shaken with an equal bulk of ether in a separator the residue on evaporation should be not much more than is left by the distilled water used.

## SACCHARUM PURIFICATUM

Commercial white sugar has usually a yellow tint, which is not objectionable provided it be very faint, or it has a tint of blue added to cover the natural yellowness—which addition is

objectionable. These added colouring matters may be more clearly noted by dissolving the sugar in its own weight of distilled water, and allowing a day or two for subsidence.

Traces of ash consisting of lime or potash salts, are usually present, and some organic matter other than sugar, but the quantity and nature of these are rarely such as to be of importance to the pharmacist if its general appearance and that of the syrup it produces are satisfactory. As cane sugar has a less active reducing power than glucose, the B.P. gives a test for the presence of the latter by heating a solution of the sugar with a little sulphate of copper and excess of caustic potash, when the presence of starch sugar would be indicated by a yellow or red precipitate of suboxide of copper being thrown down before the boiling point is reached.

### SALICINUM

Salicin, from the mode of its manufacture, might be contaminated with lead, which is used to remove tannin and colouring matters from the decoction of willow bark ; this impurity may be looked for by testing with sulphhydrate of ammonium, with which it would produce a brown tint or a black precipitate according as there is a minute or more palpable trace of lead. To cover other chances of impurity, 10 grains shaken with f3v of cold water should not entirely dissolve, but should form a clear solution with an additional drachm : the solution should be neutral and not precipitated by either tannin or carbonate of sodium. Two drachms of ether shaken with 10 grains of salicin, and filtered, should leave on evaporation a scarcely perceptible residue ; and 10 grains burned on a platinum dish should leave a scarcely perceptible ash (theoretically none).

### SANTONINUM

Santonin of commerce is usually pure, except occasionally for the presence of a little colouring matter, due to the action of

light. Two drachms of distilled water shaken with 10 grains of the sample, and filtered, should be neutral to litmus and leave not more than 0.1 grain of residue on evaporation (absence of other neutral bodies). Ten grains in f3ij Liquor Potassæ should make a clear solution. Ten grains on agitation with f3ss of chloroform makes a clear solution, which being shaken with f3ij dilute acetic acid in a separator, the acid liquor after subsidence and decantation should leave on evaporation a scarcely appreciable residue.

Ten grains powdered and mixed with f3j cold strong sulphuric acid produces a clear solution almost colourless, which, if promptly diluted with two ounces of water, deposits the santonin again as a white crystalline precipitate, and the solution separated by filtration should give no precipitate on adding ammonia in excess. The freedom from colour with the acid indicates the absence of many neutral bodies, and the absence of a precipitate with ammonia indicates its freedom from most of the alkaloids.

### SAPO ANIMALIS—SAPO DURUS

These are soda soaps, and the B.P. provides against their containing potash by requiring that, when incinerated, the ash shall not be deliquescent. This is not a probable contingency. A more likely one is the presence of free alkali, which may under some circumstances be objectionable ; hence the requirement that it shall be either neutral or very faintly alkaline to test-paper. For the majority of purposes, testing is quite unnecessary, though, if required in a liniment containing free iodine, the absence of free alkali should be insured by testing, and, if necessary, the addition of a drop or two of oleic acid till neutrality is attained.

### SAPO MOLLIS

This is a potash soap and theoretically should contain no soda, though in commerce it usually contains a trace sufficient to impart the soda yellow colour to flame ; this, however, may



be a trace too small to be of any importance. It is generally freely alkaline to test-paper, and sometimes strongly so. One drachm dissolved in an ounce of water should be rendered milky on the addition of 5 or 6 drops of dilute sulphuric acid ; if more than this can be added without producing permanent turbidity, the sample is unusually and objectionably alkaline.

### SCAMMONIÆ RESINA

The impurities to be anticipated in scammony resin are those constituents of the root which are soluble in spirit and in water, and which are present from imperfect washing ; the lumps of resin as found in commerce sometimes have moist sticky surfaces which they would not have if pure. Perfect solubility in ether excludes most of the probable impurities. The B.P. guards against adulteration with guaiacum, by requiring that its solution in alcohol shall not impart a blue colour to a slice of raw potato. A readier test to the pharmacist is to put a drop of the tincture on paper and add a drop of solution of chlorinated lime, which produces the blue colour with guaiacum. If any sample of scammony resin should not be above suspicion of adulteration, there is more fear of the presence of other resins having less characteristic reactions.

### SCAMMONIUM

Commercial scammony is a somewhat indefinite body. According to B.P. it should contain 'about 75 per cent. of resin soluble in ether ;' commercially it varies between 50 per cent. and 90 per cent.; the official standard is the lowest which should be used. The assay may be readily performed by shaking 20 grains of the powder in a corked test-tube with fʒij ether, and after repeated shakings allowing it to subside for half an hour, decanting the clear portion, repeating this treatment with a second and third portion of ether, then throwing the insoluble matter upon a small filter of double paper, washing out the tube with several small portions of ether, pouring these upon the filter till every

particle of insoluble matter is collected, then drying the filter at  $212^{\circ}\text{F.}$ , and taking the outer cone of paper as a tare for the inner; the residue should not weigh more than 5 grains. If the amount of insoluble matter is not excessive, its nature is not very important, but if the sample be unsophisticated the residue on the filter should be almost entirely soluble in cold water. Starch and earthy matters are sometimes present as adulterations; the former may be sought for in the usual way by tincture of iodine, and the latter by incinerating the filter and its contents after they have been washed with water. The ash from this quantity should be under 1 grain. A more likely adulteration of scammony, if purchased in powder, is an admixture of the resin extracted from the root. This is not easy of detection, and security against it is best obtained by buying the scammony in lump and testing its capability of making a good emulsion by simple trituration with water.

### SODA CAUSTICA

Hydrate of sodium, 'with some impurities,' chiefly water, carbonic acid, sulphuric acid, chloride, silica, and alumina; iron is also sometimes present; it is said to be as ferrate of sodium. Organic matter, too, is an occasional impurity, imparting a yellow brown colour to the solution, and sometimes nitrite of sodium, resulting from the addition of nitrate in the process of manufacture to oxidise organic matter or sulphide of sodium. The B.P. allows these altogether to amount to 10 per cent., the greater part of which should be water and carbonic acid. Commercial caustic soda, unless purchased as pure, will commonly contain more impurity than this, the carbonate, sulphate, and chloride each amounting to 4 to 5 per cent., and the hydrate of sodium being between 80 and 90 per cent. in good samples. The note appended in B.P. at the foot of the tests saying, 'Preparation containing caustic soda. Liquor Sodæ 18·8 grains in a fluid ounce,' is apt to mislead. If 18·8 grains of B.P. caustic soda be made into a fluid ounce of solution with distilled water, it would not make Liq. Sodæ B.P., but a liquor 10 per cent. deficient in strength. The B.P. requires that 40 grains of caustic soda dissolved in water

shall require about 900 grain measures of volumetric oxalic acid for neutralisation ; but as this quantity of volumetric solution neutralises 36 grains of pure  $\text{NaHO}$ , there must be about 4 grains of impurity present in 40 grains of B.P. caustic soda. Forty grains of B.P. caustic soda dissolved in water should make a neutral solution with 57 grains of oxalic acid, but if the alkali be pure it may require  $5\frac{1}{2}$  grains more. Caustic soda containing so much impurity as the B.P. allows, should not be used for making *Liquor Sodæ*, as it would be impossible to make a liquor of B.P. purity with it unless the 10 per cent. of deficiency in neutralising power were almost exclusively due to water. A good general test for pharmaceutical caustic soda is its solubility in spirit. Rub 3j of the sample in a mortar with f3ij of water, and when dissolved add an ounce of spirit (rectified or methylated), collect the precipitate on a filter and wash with spirit ; it will contain the carbonate, sulphate, silica, alumina, iron, &c., and should not exceed 2 grains. The alcoholic solution may contain chloride and nitrite. The probable presence of silica and alumina, though unimportant medicinally, must not be overlooked where the soda is used for other chemical purposes. Test for nitrite as described under 'Caustic Potash,' page 129.

### SODA TARTARATA

In the characters of this salt, the production of a yellow flame is an indication of soda, and the precipitation (of cream of tartar), on the addition of acetic acid, indicates at once potash and tartaric acid, while the free and perfect solubility in water indicates that the acid tartrate of potassium is not in excess. The salt as usually found in commerce is almost absolutely pure. The volumetric test is an unimportant one ; any little deficiency of neutralising power would almost certainly be due to the sample being slightly damp ; it is also possible, though not probable, that it might result from the presence of tartrate of ammonium, an impurity which the U.S.P. provides against. If it be desired to apply an equivalent to the volu-



metric test, 35 grains of the salt, after heating in a platinum capsule till it ceases to flame, may be thrown into hot water, capsule and all, with 15 grains of oxalic acid; the reaction with litmus should be alkaline, but should become acid with an additional grain.

### SODII ARSENIAS

The only fault to be anticipated in pharmaceutical arseniate of sodium is water in less than its due proportion. It is not desirable to trust to the correct degree of hydration of the crystals, but to dry it at the time of use and weigh it in the dry state.

The presence of arsenite of sodium—a possible impurity from faulty preparation—is according to the U.S.P. to be tested for by acidulating a solution of the arseniate with HCl and adding solution of  $H_2S$ , when the presence of arsenite would be indicated by an immediate precipitation of yellow sulphide of arsenium. The higher sulphide, resulting from decomposition of arsenic acid with  $H_2S$ , is more slowly thrown down.

A more ready examination is to dissolve a little of the arseniate with a little bicarbonate of sodium and add a drop of tincture of iodine, which will impart a yellow tint if the sample be free from arsenite, but will fail to give this colour if the latter be present.

### SODII BICARBONAS

The best commercial brands of this salt are usually very pure, and the less costly brands as a rule contain only traces of chloride and sulphate with a more or less considerable percentage of monocarbonate, and occasionally a little ammonia if made by the ammonia process now so largely practised. If the sample be from an unknown source, it is well to test it by boiling a small quantity with liquor potassæ in a test-tube and ascertaining its freedom from ammoniacal odour. This direct test will detect a smaller trace of ammonia than would be indicated by the deficiency of neutralising power under the volumetric examination as directed in B.P. The official test



with perchloride of mercury indicates that it is thoroughly carbonated if the precipitate is white. The U.S.P. is more explicit in its instruction for applying this test ; half a drachm of the bicarbonate is to be dissolved in an ounce of cold water with *very gentle agitation* and added to 5 grains of perchloride of mercury previously dissolved in ʒjss of water. This should only produce a white cloud ; neither a red colour nor red precipitate should appear within three minutes.

Heat and agitation promote the formation of this red precipitate, which after some time will appear with the most perfect samples.

The official test with perchloride of platinum would indicate the presence of either potassium or ammonium if a yellow precipitate were thrown down in the hydrochloric solution. It is probably directed against imperfectly made ammonia soda, The testing for sulphates and chlorides may both be performed on a solution of the sodium bicarbonate in a slight excess of dilute nitric acid, and with first-class brands there is usually no precipitate with either chloride of barium or nitrate of silver.

### SODII BROMIDUM

The impurities to be sought for in this salt are carbonate, chloride, and iodide of sodium. If carbonate be present, it will precipitate with chloride of barium, and the precipitate will redissolve on the addition of HCl. Chlorine and iodine should be tested for as described under 'Bromide of Potassium.' Ten grains of bromide of sodium in 1 ounce of water, if precipitated with sixteen grains of nitrate of silver, should yield a further precipitate on the addition of one grain more of the nitrate, after which a further addition should not produce a further precipitate. This test is practically equivalent to the B.P. volumetric test, and the obtaining of the required result does not prove the purity of the salt. If the precipitation is complete with less than the normal quantity of the nitrate, the impurity may consist of moisture (most probably) or iodide. If more than normal quantity be required, chloride

may be present, and if exactly the normal quantity be required, all or none may be present.

In testing bromide of ammonium the pharmacopœia directs the addition of a small quantity of chromate of potassium to the bromide, in which case the point at which the complete precipitation of the bromide takes place is indicated by the commencement of the precipitation of red chromate of silver ; this is in some respects more convenient than observing that a further addition of nitrate does not throw down more bromide of silver, and it is equally applicable to the testing of the bromides of potassium and sodium.

### SODII CARBONAS

The common commercial carbonate of sodium always contains sulphate and chloride, but that which has been re-crystallised for pharmaceutical use is nearly pure. The B.P. allows traces of chlorides and sulphates, and by volumetric analysis it allows 4 per cent. of deficiency in neutralising power. If such deficiency exist in the presence of only traces of chlorides and sulphates, the most probable impurity is water. It is rather difficult to get the crystals, which are produced on the small scale, deprived of adhering water without some loss of water of crystallisation, and a more probable fault is deficiency rather than excess of water. The ordinary impurities in the quantities in which they are usually present are of no importance in relation to any of the purposes for which this salt is used in pharmacy.

### SODII CARBONAS EXSICCATA

Not subject to any impurity of importance to pharmaceutical uses.

### SODII CHLORIDUM

Chloride of sodium, if purchased under its chemical name, should be pure, but if bought under its official synonym of

'common salt' it will generally be found to contain chlorides of calcium, magnesium, and sometimes aluminium in appreciable quantities, and at least traces of sulphate, &c. The earthy impurities will be thrown down by the addition of carbonate of sodium. When applied only to the uses indicated in the Pharmacopœia, the earthy impurities are of no moment, but if prescribed along with ammonia, as it sometimes is for a lotion, a pure salt is desirable.

### SODII HYPOPHOSPHIS

From the official mode of making this salt (if such be followed by the manufacturer) it would be liable to contamination with carbonate of sodium or hypophosphite of calcium. These are provided against in the B.P. by requiring that there shall be no effervescence with acids, and only traces of precipitate with oxalate of ammonium. Phosphate of sodium may be present as a result of oxidation ; its presence would be indicated by the official test with acetate of lead, but as sulphates and carbonates would give a similar reaction, phosphate should only be assumed to be present if other tests for sulphate and carbonate give negative results. The oxidation test, as given in the Pharmacopœia, is simple, easy, and requires no modification to adapt it for use at the dispensing counter.

### SODII IODIDUM

Chloride, bromide, iodate, and carbonate of sodium are the impurities to be anticipated. Of these the iodate is the most objectionable and probably the most frequent ; it is indicated by tartaric or other non-oxidising acid liberating iodine which if present in any quantity worth noting is sufficiently indicated by the yellow brown colour of the solution, without the aid of starch which the pharmacopœia directs. Chloride is detected by precipitating with an excess of nitrate of silver, which throws down any chloride present along with the iodide ; when this precipitate is treated with solution of

ammonia, the chloride is dissolved, leaving the iodide insoluble ; if a portion of clear solution be filtered off, and treated with an excess of nitric acid, any chloride present is again precipitated. Carbonate would be indicated by a solution giving a precipitate with lime-water or saccharated solution of lime as the Pharmacopœia directs, or, if the quantity be large, by alkalinity to litmus paper, or effervescence with acids. Ten grains of the iodide should, when precipitated with eleven grains of nitrate of silver, yield a further precipitate on adding one more grain of nitrate, but after this a further addition gives no further precipitate. Precipitation with the second additional grain of nitrate would suggest the presence of chloride or bromide.

### SODII PHOSPHAS

Sulphate of sodium is the most likely impurity in this salt, and is indicated if a portion of the precipitate thrown down by chloride of barium be insoluble in dilute nitric acid, the phosphate of barium being soluble in this acid. If strong nitric acid were used, the sparing solubility of nitrate of barium or phosphate of barium in the strong acid might lead to the supposition that sulphate was present.

### SODII SALICYLAS

The impurities to be anticipated are carbonate of sodium, salicylic acid, and organic matters other than salicylic acid. The complete solubility in double its weight of rectified spirit indicates the absence of carbonate and sulphate of sodium, and the free solubility in about its own weight of water shows the absence of uncombined salicylic acid.

The testing of this salt for chlorides and sulphates is directed to be performed with the aid of sufficient spirit to redissolve the salicylic acid which is precipitated when an aqueous solution of the salicylate is acidulated with nitric acid. The use of spirit is not essential, but it adds to the delicacy of the reaction, and is quite as convenient as filtration, which would



otherwise be necessary before adding the nitrate of silver or chloride of barium solutions. The solubility of the salicylate of sodium in cold strong sulphuric acid without effervescence, shows its freedom from carbonate ; and the solution not being coloured indicates the absence of many organic impurities with which it might possibly be contaminated.

### SODII SULPHAS

Sulphate of sodium of commerce is a salt which scarcely requires examination. If in dry clear crystals, neutral to test paper, its purity may be assumed. Being an efflorescent salt, deficiency of water is the point in which it is liable to deviate from its normal condition, but from the mode of its manufacture it may possibly contain also carbonate of sodium, chloride of sodium, or sulphuric acid. The former would be indicated by an alkaline reaction, or by a solution of the salt giving a precipitate with lime water ; the latter by an acid reaction, and chloride by its precipitating with nitrate of silver.

### SODII SULPHIS

Sulphite of sodium commonly contains traces of sulphate, and may contain also carbonate or hyposulphite. An aqueous solution (3ss in 3ss) should not effervesce with dilute hydrochloric acid (freedom from carbonate), should not become milky (freedom from hyposulphite), should have a strong sulphurous odour, should dissolve 3ss of iodine forming a colourless solution, but should become brown on the addition of another grain. The quantity of sulphurous acid and water may both vary in relation to the soda, and from either or both of these causes the action upon iodine may be in excess of the theoretical proportion. The B.P. does not give any quantitative indication except the formula  $\text{Na}_2\text{SO}_3, 7\text{H}_2\text{O}$ . A little latitude would be reasonable.

## SODII SULPHOCARBOLAS

The probable impurities in this salt, if prepared by the official process, are either sulphocarbolate of barium or carbonate of sodium, according as either of these salts might be used in excess. The B.P. test with  $\text{BaCl}_2$  would indicate the presence of either carbonate or sulphate if an immediate precipitate took place, and either of these would be less objectionable than a contamination with the barium salt, the absence of which would be indicated by the appearance of this precipitate.

## SODII VALERIANAS

There is little liability to impurity in valerianate of sodium, except an excess of soda either as caustic or carbonate. Perfect solubility in rectified spirit and freedom from any clearly marked alkaline reaction are sufficient to secure this point. Butyrate of sodium may be present from imperfect rectification of the amylic alcohol from which the valerianic acid has been produced. The B.P. does not notice this under the head of valerianate of sodium, but under the tests for valerianate of zinc it requires that a distillate with sulphuric acid should not precipitate with acetate of copper. (See 'Zinc Valerianas.')

## SODIUM

Sodium of commerce, if scraped free from the superficial coating of oxide, may be regarded as a pure chemical. Any examination of its neutralising power is quite uncalled for.

## SPIRITUS ÆTHERIS NITROSI

For many years it has been a difficulty to provide a satisfactory test for the quality of spirit of nitrous ether, and no less a difficulty to provide for dispensing a spirit of nitrous ether answering the tests officially required. This has been altered in a great degree in the B.P. of 1885, in which the test has

been settled upon and the process made to work up to it. The official name implies that it is a solution of nitrous ether in spirit. The official definition and test imply that it shall contain nitrous compounds. It has been shown that a compound can be produced answering to the B.P. test which does not contain any nitrous ether ; it cannot, however, claim to be B.P. spirit of nitrous ether unless it contains those nitrous compounds naturally resulting from the B.P. process.

On the other hand a test may be devised which will give, with pretty close approximation, the same estimate of nitrous compounds as the official test without involving so much trouble to the pharmacist, and the indications from which may be of equal value, though not necessarily expressed in volumes of gas. In the official test the volume of gas liberated is the mode adopted to indicate the quantity of nitrous ether ; but if the quantity of a reagent required to produce a definite reaction can give us the same information, there is no reason why we should not consider it equally satisfactory.

As the B.P. does not give particulars of the test which it recommends, it will be convenient here to describe the apparatus and mode of use before giving an account of the process which I would suggest as more convenient to the pharmacist and sufficiently accurate to show whether or not his sample comes within the somewhat liberal latitude which the authorities sanction.

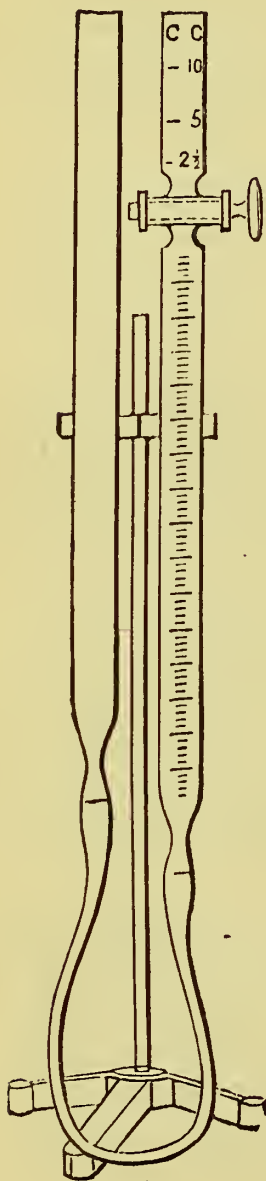
The nitrometer is an instrument which was originally devised by Dr. Lunge for estimating nitrous compounds in crude oil of vitriol, and has been modified in several small particulars to adapt it for other purposes. The following is known as Allen's nitrometer, and the official reference is to Mr. Allen's paper describing its use. When used for testing spirit of nitrous ether, the stop-cock being open, brine, which should be at least a nearly saturated solution of common salt, is poured into the plain tube, which is connected by an india-rubber tube with the graduated tube ; the former is then raised till the brine rises in the latter, exactly to the stop-cock, which is then closed, and the plain tube is lowered again till the level of the brine in it is

a little below the cock. One volume of the sample to be tested is poured into the graduated cup above the cock. A volume may be any convenient quantity, but, as Allen's instrument is

graduated into c.c., five c.c. may be taken as a convenient volume ; the cock is then to be cautiously opened till all the spirit of nitrous ether has passed into the tube below the cock without allowing any air to enter. An equal volume of strong solution of iodide of potassium (about 30 per cent. is a suitable strength) is then to be run in in the same manner ; but this requires more caution than the former, as the spirit usually contains so much free acid as to start a considerable effervescence directly the iodide comes in contact with it, and there is a risk of some of the gas bubbling up through the cock unless the brine tube be sufficiently lowered, and if the level of the brine in it be much below the level in the graduated or gas tube there is the greater risk of admitting air. It is, therefore, best to close the cock before the last drop of the solution has run through ; then pour into the cup an equal volume of dilute sulphuric acid, and let it run in with the same caution, always keeping the level of the liquor in the brine tube a little below that of the liquor in the gas tube. When the acid has been added and the cock closed, the gas tube may be shaken laterally, so as to mix the samples with the reagents thoroughly without mixing them with the bulk of the brine. The effervescence is over in a few

minutes ; the brine tube is then adjusted so that the level of the liquid in it corresponds with that in the gas tube, and the volume of the evolved gas is read off, which, if the sample were a newly made one, should amount to 35 c.c.

The liberation of the nitric oxide gas is accompanied by





the liberation of an equivalent of iodine, which may be estimated, according to B.P. method, by hyposulphite of sodium, if the nitric oxide be first got rid of, which may be done by raising the brine tube and opening the cock till the gas is expelled, but taking care that no liquor comes above the cock. The ruddy fumes should be blown away before running out the brine, otherwise more iodine would be liberated ; but having got rid of these, the whole contents of the nitrometer may be run into a dish, the nitrometer washed out with water, and the quantity of iodine contained in the brine and washings ascertained by titrating with hyposulphite. The results should agree nearly with those indicated by the volume of nitric oxide. The sources of discrepancy between the results by volume of gas and by titration are first, that a small quantity of the gas remains in solution, so diminishing the bulk measured ; then the dissolved gas absorbs oxygen when exposed to the air, and oxidises a further portion of the hydriodic acid contained in the brine, due to the decomposition of the iodide of potassium by the sulphuric acid, these being necessarily in excess of the quantity requisite to decompose the ethyl nitrite. The estimation by volume of gas may thus indicate a little less than the true amount of the nitrite, and the estimation by hyposulphite is apt to be a little in excess of the truth.

In the process which I devised for a dispensing-counter test I endeavoured to diminish, as far as was practicable in a rather rough operation, these two sources of error. The process is as follows :—

A solution of hyposulphite of sodium is prepared of such a strength that the reaction is completed with a fluid drachm of spirit of nitrous ether a little above the normal strength ; and having ascertained how much of the sample under trial completes the reaction, calculate the relation which the sample bears to the normal. For this purpose it is convenient to prepare the solutions thus :—

1. Hyposulphite of sodium	.	.	.	4 grains	.
Chloride of sodium	.	.	.	40	„
Iodide of potassium	.	.	.	20	„
Water	.	.	.	2 drachms	

2. Spirit of nitrous ether (the sample to be tested) . . . . . 2 fluid drachms  
 Dilute sulphuric acid . . . . . 1 „, drachm

Place the former in a shallow dish—the lid of a three or four-ounce ointment-pot is suitable—add to this f3jss of No. 2 (i.e. containing f3j sp. æth. nit.), stir well and blow away the ruddy fumes as fast as they form on the surface of the effervescing liquor. If the solution has acquired and retained a brown colour, it will indicate that the f3j of spirit of nitrous ether which is contained in this addition would have evolved more than 7 volumes of gas (i.e. about 7·4 fluid drachms) had it been tested in the nitrometer by the B.P. process. If, however, the brown colour was not permanent, the No. 2 solution may be added in the like manner, f3ss at a time, with stirring and blowing off the ruddy fumes till the brown colour is permanently developed. As each addition of f3ss means 20 minims of the spirit of nitrous ether, the quantity required to evolve 7·4 fluid drachms of nitric oxide gas is ascertained, and from this datum the volumes of gas liberated by one volume of the spirit may be calculated. Thus, if the first f3jss of No. 2 gives a brown tint, we may say the sample gives 7·4 volumes or more.

$$\begin{aligned} \text{f 3ij being required} &= 5\cdot5 \text{ vols.} \\ \text{f 3ijss „ „} &= 4\cdot4 „ \\ \text{f 3iij „ „} &= 3\cdot7 „ \end{aligned}$$

The first would be rather over strength, the second a fair average strength; the third would be on the debateable ground left doubtful by the official statement that it is to yield ‘not much less than five volumes;’ the fourth, we would say without hesitation, was below the strength which should be sanctioned.

The points about which care is required in the performance of this test are to avoid loss of nitrous ether vapour by evaporation, and to avoid continued contact of the nitric oxide gas with the air and with the solution containing the iodide, as the nitric oxide combines with oxygen from the air, and yields it up readily to the hydriodic acid which has been liberated from

the iodide of potassium by the action of the sulphuric acid. This latter source of error is sought to be avoided by the addition of chloride of sodium to the No. 1 solution—nitric oxide being less soluble in brine than in water—and by operating in a shallow dish and blowing off the gas as it is liberated.

The loss by evaporation of nitrous ether is sought to be avoided by making the additions of the spirit of nitrous ether to an excess of the solution of iodide and hyposulphite until the terminal reaction is reached. Probably neither of these sources of error is entirely removed ; but they act in opposite directions and tend to compensate for one another. I have found the results agree more closely with the indications of the nitrometer than the results obtained by various modifications of this test which I have tried in the hope of finding some mode of operating which should be equally simple and not involve this somewhat circuitous mode of calculating the results.

This process makes no pretence to scientific accuracy, but readily indicates whether or not the sample under examination would evolve so much gas as comes within the limits fixed by the authorities. Or, if the percentage of nitrite of ethyl be defined, as has been recently suggested, the 7·4 volumes correspond to 2·6 per cent., and 3·7 volumes equal 1·3 per cent., this latter being as high as might be expected for an article not sold as spiritus ætheris nitrosi, but as sweet spirit of nitre.

### SPIRITUS AMMONIÆ AROMATICUS

This is liable to vary in the strength of the spirit, and in the proportion of caustic ammonia and carbonate of ammonium which it contains. Its sp. gr. is defined to be 0·896 ; a variation in the third decimal figure is reasonably allowable. Two fluid drachms coloured with litmus and added to 8 grains of oxalic acid, dissolved in half an ounce of water, should retain its blue colour when heated to drive off the carbonic acid, but should become permanently red on adding one grain more of the acid.



Two fluid drachms on the addition of 8 grains of chloride of barium, dissolved in half an ounce of water, gives a copious precipitate, the filtrate from which gives a further precipitate with more of the chloride, otherwise too small a proportion of the ammonia is in the condition of carbonate.

### SPIRITUS RECTIFICATUS

Spirit is to be tested for its strength and freedom from fixed or empyreumatic impurities. The sp. gr. should be 0·838, and this is all that is required to test its strength if at the same time its freedom from abnormal odour is satisfactory. Methy-  
lated spirit, or spirit containing other impurities, might have the right density, and it is possible that it might also have the right odour and flavour and still not be a good spirit, but the pharmacist is greatly protected by the excise laws, and there is scarcely a possibility of a spirit appearing in commerce which has the correct odour, flavour, and density, and is not correct for pharmaceutical use. Wood spirit may be so far purified that it would cease to be noticeable if used as an adulterant of grain spirit, but if purified so as to become potable it is subject to the same duty as other potable spirit, and its cost, including duty, would put it out of competition with grain spirit. The customary test, and the most convenient one for methylic alcohol in rectified spirit, is to mix with it an equal volume of liquor potassæ, when a darkening of the colour would take place if methylic alcohol were present ; as the brown tint observed in this reaction may be produced by aldehyde and some other impurities, the presence of the colour is not proof of the presence of methylic alcohol, though the absence of colour after standing some hours is evidence of the absence of this impurity as well as the absence of aldehyde, tannin, &c. Some other tests for methyl alcohol are more characteristic, but too troublesome for dispensing-counter use. The B.P. test with nitrate of silver is intended to exclude more than traces of fusel oil and other organic impurities ; half a grain of the nitrate being dissolved in 4 ounces of rectified spirit, and



exposed to bright light for a day, produces a black precipitate of reduced silver, but the spirit decanted from this precipitate should not give any further reaction on adding more of the reagent and repeating the exposure to light. Pure alcohol does not reduce nitrate of silver.

### STRYCHNINA

Crystallised strychnine, as now found in commerce, is usually a very pure chemical. Its solubility in strong sulphuric acid without coloration indicates the absence of some of the amorphous matters of the *nux vomica* with which it might be contaminated, and the absence of a red coloration when treated with nitric acid proves its freedom from brucine. If any further examination were desirable, its solubilities give general indications of purity if tested as follows:— 1 grain of the strychnine shaken with 1 drachm of ether and settled, the ether evaporates with scarcely visible residue. One grain shaken with a drachm of water and a drop of acetic acid, forms a clear solution which affords a copious precipitate on adding a drop of strong solution of ammonia; now add a fluid drachm of chloroform, and shake the mixture; the precipitated strychnine immediately dissolves in the chloroform, and when this has subsided, the aqueous solution decanted will evaporate with a scarcely appreciable residue at a temperature below 212° F.

### SULPHUR

The only common impurities of commercial sulphur are sulphuric acid and sulphate of lime, the former in the unwashed flowers of sulphur, and the latter in the precipitated sulphur known as milk of sulphur. Selenium and sulphide of arsenium are said to be common impurities, though in quantity too small to be readily detected.

The tests desirable to be applied are volatilisation, to prove its freedom from fixed matters including sulphate of lime, and total insolubility in cold liquor of ammonia; 3j of the sulphur shaken with 3ij solution of ammonia and 3ij water and filtered; the fil-

trate on evaporation should leave no fixed residue ; the presence of a residue might indicate sulphuric acid or sulphide of arsenium.

### SULPHURIS IODIDUM

This is rather a loose combination of sulphur and iodine ; the sulphur may be in excess of its legitimate proportion. Forty grains of the sample with two ounces of water in an open beaker or dish, boiled till it no longer smells of iodine, should leave 8 grains of sulphur. Additions may be made to the water to avoid its boiling to dryness before the odour of the iodine has gone, but when this is accomplished, evaporation may be continued to dryness, and the weight of the residue ascertained. Any excess over 8 grains may be either too large a proportion of sulphur, or some other impurity.

### THERIACA

The old form of treacle was usually contaminated with iron, sometimes to an objectionable extent. With the improvements in sugar manufacture this impurity has nearly disappeared. If to be used only as a pill excipient, testing is of no moment, but for other purposes the detection of iron may be readily accomplished by diluting the treacle with 5 or 6 times its bulk of water and adding a little yellow prussiate of potash, when a blue coloration will be produced if iron be present.

### THYMOL

The first requirement of thymol, like volatile oils, is that it should have the true odour of the plant from which it is obtained, but while we say the true odour we cannot say the full odour ; an odour may be subtracted, but no odour must be added.

It is sometimes difficult to judge whether the difference between the proximate principle and the entire plant is due to addition or subtraction. Thymol, though it has an odour of thyme, has not the whole odour. Its density is so closely that of water that it sinks in cold water, but, expanding as it melts,

it floats when fluid at a temperature of  $110^{\circ}$  to  $125^{\circ}$  F. The boiling and melting points of essential oils vary much with sundry circumstances, with fractionation,<sup>1</sup> &c., but when reduced to proximate crystalline principles, such as thymol, these points should cease to vary, and the latitude allowed by the B.P. in the melting-point, i.e. from  $110^{\circ}$  to  $125^{\circ}$  F., may be supposed to allow for the presence, more or less, of the other oily principles of lower freezing points. If any considerable proportion of the less crystallisable oils be present, it would become evident on dissolving the thymol in liquor of potash, when the more fluid oils rise to the surface. There is little probability of thymol failing to respond to the official requirement that it shall dissolve freely (though not in all proportions) in alcohol, and shall entirely volatilise at  $212^{\circ}$  F. With the view of distinguishing thymol from other stearoptenes the B.P. gives a colour reaction to be obtained by dissolving f3j thymol in f3ss glacial acetic acid, adding f3jss of strong sulphuric acid and warming the mixture, when a reddish-violet colour should be produced. This is not more characteristic than the odour, nor is it necessary to be tried if the odour be correct.

### UNGUENTUM HYDRARGYRI

Mercurial ointment is one of the few ointments that the ordinary pharmacist does not make for himself, and consequently one the correctness of which it is desirable he should have the means of verifying. Its quality may be judged most conclusively by dissolving the grease in ether and obtaining the mercury in the fluid state, but this is not very easily done, as the mercury retains its pulverulent condition rather persistently, and this is especially the case if the mercury has been mixed with the aid of sulphur, as the B.P. directs under the instructions for mercurial plaster, a custom which is practised by some makers. The density of the ointment gives a fair presumptive evidence of its strength, especially if corroborated

<sup>1</sup> *i.e.* separation into fractions by interruption of the distillation at one or more points of the process.



by an examination with iodine. Taking the sp. gr. of the fatty matter as  $\cdot 9348$ , and that of the mercury as  $13\cdot 6$ , the theoretical gravity of the ointment should be  $1\cdot 71$ , but from the nature of its manufacture there are always minute air-bubbles diffused through its mass which reduce this figure more or less. Practically I have found its sp. gr. to be  $1\cdot 655$  when taken in the following manner. Take a little compact piece, about 8 or 10 grains, out of the bulk, roll it into the form of a pill by the aid of a wet finger on a wet surface of paper or wood, throw it into some dense aqueous solution in which it floats, dilute this with gradual additions of water till the ointment and solution have the same density, and then take the density of the solution with the sp. gr. bottle. There are but few pharmaceutical liquors sufficiently dense for this purpose. Sulphuric acid is too corrosive, and evolves too much heat on dilution to be convenient, but a saturated solution of chloride of zinc or a syrupy phosphoric acid of sp. gr.  $1\cdot 750$  may be conveniently used.

### VINUM AURANTII

Orange wine should contain 10 or 12 per cent. of alcohol according to B.P., but no instruction is given for the estimation of the alcohol, which cannot be done by simply taking its gravity, as this is so much influenced by the quantity of sugar, &c., which it may contain. A correct estimate of its alcoholic value may be obtained by submitting it to distillation and taking the density of the distillate. A distillatory arrangement may be extemporised thus :—Take a couple of 10-ounce flasks connected by a glass tube bent twice at right angles, place 6 fluid ounces of the wine in one and fit the tube to it with a good tight-fitting cork, the end of the tube not passing far down the neck of the flask ; the other end of the tube should pass down to about the middle of the other flask, which is to act as a condenser, and should be held in place by a loosely fitting cork ; the flask containing the wine is to be heated by a salt-water-bath or other gentle source of heat, and the condensing flask is to be kept cool by being covered with wet cloths, and kept standing



in a dish of cold water. If the distillation be conducted slowly in this fashion, the alcohol will pass over without appreciable loss in the first 3 ounces, and if this be made up to 6 fluid ounces with distilled water, and the density taken, a reference to a table of spirit densities will now indicate correctly the percentage of alcohol in the wine. The density thus obtained for orange wine should be about '980 to '984. A fair approximation may be obtained with less trouble, by taking the density of the wine without distillation, then boiling a given quantity till the alcohol is driven off—say the bulk reduced to one-half—then adding water to bring it up to its original bulk, and again taking its density, which will now be slightly higher than water, and subtracting this excess of density from that obtained for the original wine; the difference will give the indication of alcoholic strength; thus, if the wine had a gravity of '988 in its original state, and a gravity of 1'004 after boiling and making up to measure, the excess of the latter over water is 0'004, and this, subtracted from 0'988, gives 0'984, which indicates 10 per cent. of alcohol. See table under 'Alcohol.'

### VINUM XERICUM

The above methods are of course applicable to sherry, which by this treatment should give the ultimate figure as about 0'975 or 17 per cent. of alcohol.

### ZINCI ACETAS

The chief impurities to be anticipated in acetate of zinc are iron, copper, lead, and sulphuric acid. The salt is simply tested by its solubility in excess of ammonia, and by the whiteness of the precipitate which the ammoniacal solution yields with sulphhydrate of ammonium. Small quantities of ferrous iron or lead are the only probable basic impurities which would escape detection by the former reagent, and they would be clearly indicated by the darkness of the precipitate yielded with the latter. Sulphuric acid would be detected by chloride of barium as usual.

## ZINCI CARBONAS

Carbonate of zinc may contain iron, in which case its tint will not be purely white, but inclining to yellow. If copper be present its tint may incline to blue. It should be entirely soluble in liquor of ammonia, and the solution should give a pure white precipitate with sulphhydrate of ammonium. Lime and magnesia are proved absent by the solubility in ammonia, and the whiteness of the precipitated sulphide excludes iron, copper, and lead. If the carbonate has been imperfectly washed it may be contaminated with sulphate of zinc, or sulphate or carbonate of sodium. To detect these let a drachm of the sample be shaken with an ounce of distilled water and thrown on a filter; a drop of the filtrate should evaporate on a clean slip of glass with a scarcely noticeable residue.

## ZINCI CHLORIDUM

Chloride of zinc may contain iron and lead, and invariably contains some oxide, or oxychloride of zinc; the latter does not condemn the sample though the former would.

The sample, when treated with two or three times its weight of water, makes a turbid solution, the insoluble being of considerable bulk though of small weight. It should entirely dissolve in excess of solution of ammonia, and the solution thus obtained should give a white precipitate with sulphhydrate of ammonium. Very small traces of iron, copper, or lead might escape detection in this way from the large bulk of sulphide of zinc through which it would be diffused. Such small traces of copper or lead would not escape detection if an acid solution of the zinc chloride were treated with sulphide of hydrogen, as there would then be no zinc precipitated to mask the blackness of the other sulphides, but iron is not precipitated in the acid liquor, and would be required to be sought for by testing with the ferro- and ferricyanides of potassium. The fused chloride of zinc of pharmacy rarely

demands any elaborate examination. Chloride of zinc supplied in solution would be more open to suspicion, and might contain chlorides of the alkaline or earthy metals. If it be desired to examine for these, the solution should be treated with an excess of ammonia, and then sulphhydrate of ammonium in excess, and filtered (the filtrate should have an odour of the sulphhydrate, or must be tested with more to insure its excess); the clear filtrate on evaporation to dryness will leave no fixed residue if the zinc chloride be pure. A fixed residue might contain chlorides of any or all of the following metals: calcium, aluminium, magnesium, potassium, and sodium.

### ZINCI OXIDUM

Oxide of zinc of pharmacy is usually of satisfactory chemical purity, though mechanically it is often wanting in that impalpable smoothness which is essential to the production of good zinc ointment. Indeed, we may say freedom from grit is more important than freedom from any of the chemical impurities which are likely to be present. Oxide of zinc may be tested for impurities by the same methods as the carbonate, except that in testing its solubility in ammonia a solution of carbonate of ammonium should be used instead of liquor of ammonia, as the latter has but little action on anhydrous oxide of zinc. Or the tests may be applied to a portion thrown into hydrochloric acid which should dissolve entirely without effervescence, and the solution should give the same reactions as described under chloride of zinc.

### ZINCI SULPHAS

Sulphate of zinc is liable to the same impurities as the acetate, except that lead is not to be anticipated, and iron is much more probable. The B.P. directs a special test with tincture of galls, which gives an inky tint with iron. If this test be not applied, the production of a pure white precipitate with sulphhydrate of ammonium would indicate at once the



absence of both iron and copper. A trace of iron too small to be detected by tincture of galls cannot be regarded as of any importance in pharmacy. Another official test for iron is to secure the peroxidation of the iron by boiling with a little nitric acid, after which an excess of solution of ammonia would dissolve the zinc oxide at first precipitated, but would leave the ferric oxide. Upon the same principle in the manufacturing process, chlorine water is directed to be added to oxidise any ferrous salt, and thus facilitate its precipitation with carbonate of zinc. A manufacturer would more conveniently use chloride of calcium instead of chlorine water, and might thus introduce traces of lime and chlorine into the product ; but from the very sparing solubility of sulphate of calcium, and the exceeding solubility of chloride of zinc, these would be effectively separated in the process of crystallisation, and, if present at all, would be in too small quantity to be of importance. The chlorine may be examined for as usual with nitrate of silver, and the lime by adding oxalate of ammonium to a solution of the zinc salt in excess of ammonia, oxalate of zinc being soluble in ammonia ; a white precipitate would indicate lime. The sulphates of magnesium and sodium, though not probable impurities in sulphate of zinc, might, from the similarity of their crystalline form, be present as accidental admixtures or substitutions, and would be detected by the solution of the sample treated with excess of ammonia, and then with excess of sulphhydrate of ammonium, giving a filtrate which on evaporation would give a fixed residue ; the sulphate of ammonium being volatilised below red heat, any other alkaline or earthy sulphates would be left.

### ZINCI SULPHOCARBOLAS

This salt is more likely to be at fault as regards its acid than in respect to its base, though if made by decomposition with the sulpho-carbolate of barium or calcium, these latter bases might be present and would be detected by a precipitate being thrown down by oxalate of ammonium. Sulphate of zinc is a not improbable impurity, and would be indicated, if in



considerable quantity, by being insoluble in rectified spirit, or, if traces only, by the usual reactions with chloride of barium giving an immediate precipitate.

### ZINCI VALERIANAS

This salt, if prepared by decomposition of valerianate of sodium with sulphate of zinc, may contain sulphate of sodium from imperfect washing of the crystals. The presence of basic impurities is best detected by dissolving the salt in excess of ammonia, precipitating with excess of hydrosulphate of ammonium, filtering, evaporating the filtrate, and igniting the residue, which should entirely volatilise below red heat.

Butyrate may also be present if the sodium salt from which it has been prepared contained this impurity. It is to be detected by testing with acetate of copper; the butyrate of copper, being a sparingly soluble salt, is thrown down as a precipitate if a saturated solution of acetate of copper be added to a saturated solution of valerianate of zinc, if the latter be contaminated with butyrate of zinc. This is the test adopted by the U.S. pharmacopœia. The B.P. directs that the acid or acids of the zinc salt shall be liberated by sulphuric acid, and separated by distillation, the acetate of copper being added to the distillate. I do not know that the B.P. method has advantages sufficient to compensate for its greater complexity.

# INDEX

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SALTS as a rule are indexed under their bases only ; a few exceptions will be found, such as bichromate, hyposulphite, permanganate, &c., when the acid radicle is the active agent in a test.

Substances in which there is a close resemblance between the English and Latin names will generally be found under one of these only ; thus " Acid, acetic " does not appear again as " Acidum aceticum." But " adeps " is indexed also as " lard," " argentum " as " silver," &c.

Cross references have been used whenever they appeared desirable ; sundry of the tests are indexed repeatedly when variations in the mode of use occur.

Reagents are occasionally indexed, where they are used, if any modification of the mode of their use is indicated.

English names are indexed where the subject is habitually treated in English ; thus " Estimation of mercury " does not appear under " Hydrargyrum."

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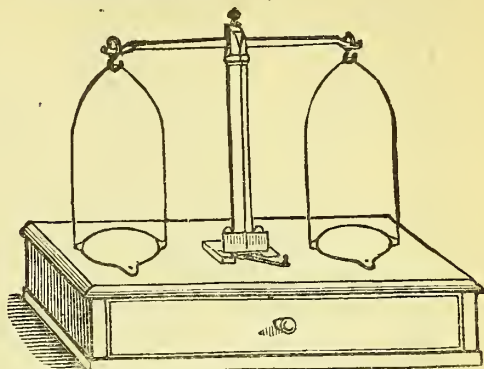
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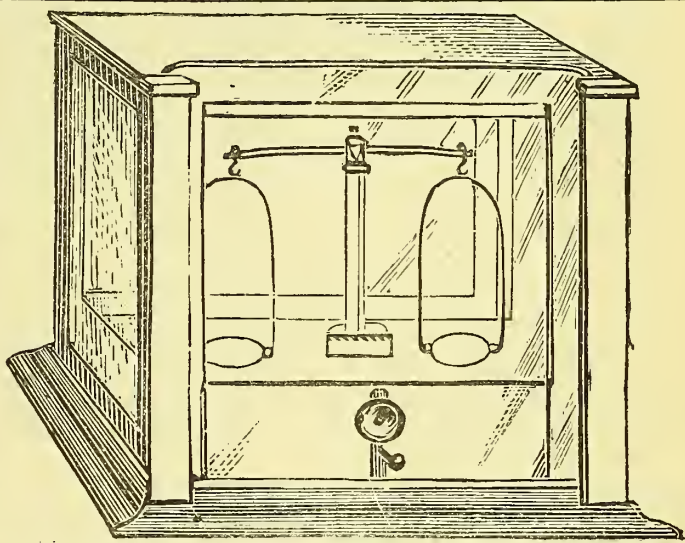
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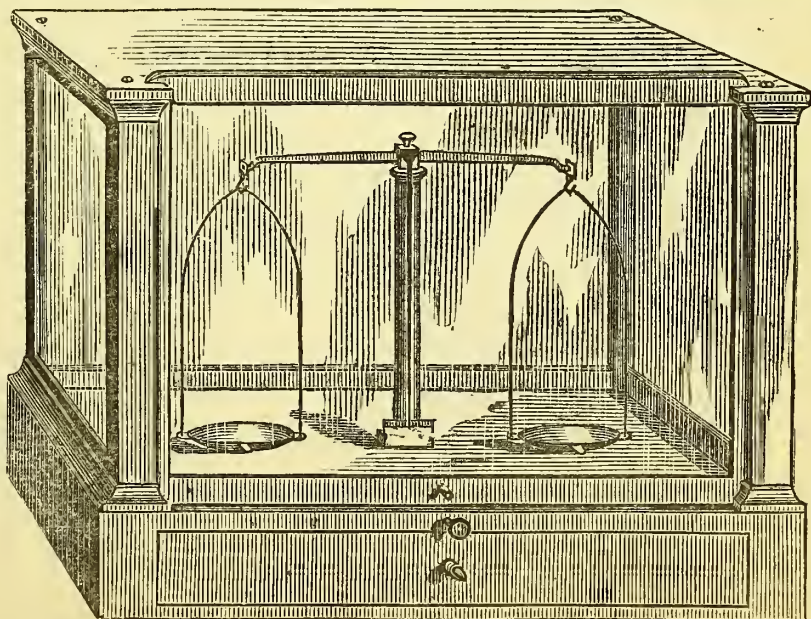
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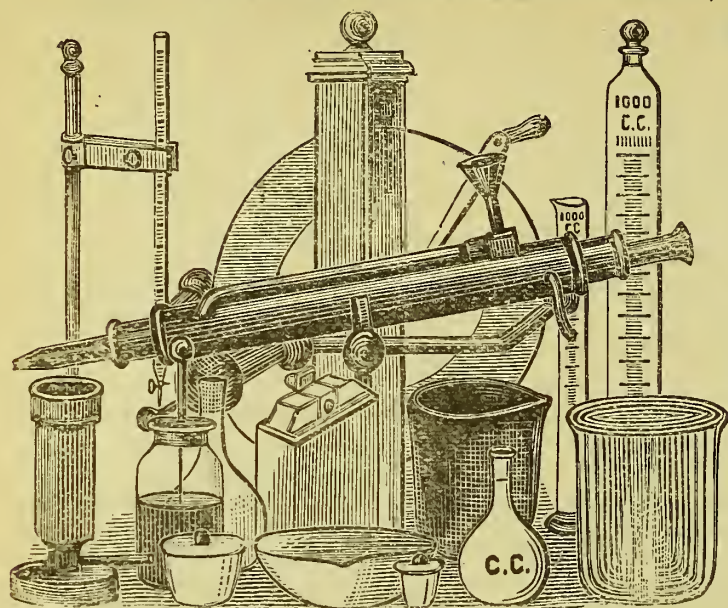


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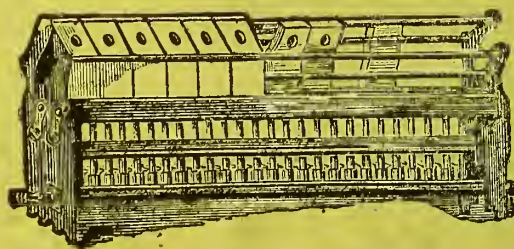
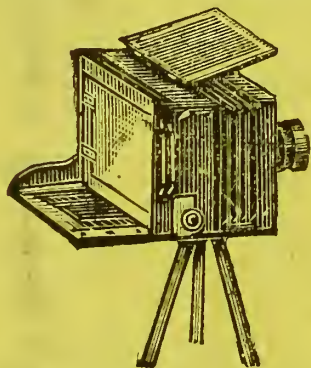
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